ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL OF SCIENCE ENGINEERING AND TECHNOLOGY

INVESTIGATION OF THE EFFECT OF CARBON NANOTUBES ON HYBRID GLASS/CARBON FIBER REINFORCED COMPOSITES

M.Sc. THESIS

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İSTANBUL TEKNİK ÜNİVERSİTESİ ★ FEN BİLİMLERİ ENSTİTÜSÜ

KARBON NANOTÜP KULLANIMININ KARBON/CAM HİBRİT KOMPOZİTLER ÜZERİNDEKİ ETKİSİNİN İNCELENMESİ

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Shahrukh Shahbaz, a **M.Sc.** student of ITU **Graduate School of science Engineering and Technology** student ID **503131814**, successfully defended the **thesis** entitled "INVESTIGATION OF THE EFFECT OF CARBON NANOTUBES ON HYBRID GLASS/CARBON FIBER REINFORCED COMPOSITES", which he prepared after fulfilling the requirements specified in the associated legislations, before the jury whose signatures are below.

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To my beloved parents and spouse,

FOREWORD

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TABLE OF CONTENTS

ABBREVIATIONS

CNTs : Carbon Nanotubes
PAN **:** Poly Acrylonitrile
SWCNT **:** Single walled Carbon Nano Tubes
MWCNT **:** Multi Walled Carbon Nano Tubes
CCC **:** Carbon-Carbon- Carbon
GGG **:** Glass-Glass-Glass
CGC **:** Carbon-Glass-Glass
GCG **:** Glass-Carbon-Glass
DMF **:** Dimethyl Formamide
UTM **:** Universal Tensile/Testing Machine

LIST OF TABLES

Page

LIST OF FIGURES

Page

INVESTIGATION OF THE EFFECT OF CARBON NANOTUBES ON HYBRID GLASS/CARBON FIBER REINFORCED COMPOSITES

SUMMARY

Composites have been attaining great concentration due to the customized properties of reinforcements and matrices and their ability to make high performance materials to replace the current structural materials. The development for the high performance composites with very low weights of materials is keeping high interest levels for the researchers. Few decades later, the discovery of Carbon Nanotubes helped the researchers in making a way for the high performance materials. This discovery also took a positive effect on the performance of composite materials. Carbon Nanotubes have been acknowledged widely for strengthening the mechanical properties of the composites. There are several ways to accommodate the interaction of the CNTs within the composite materials but the costs are high. Chemical Vapour Deposition is an effective way to grow CNTs on reinforcement but it requires higher temperatures and time. The elevated temperature also affects the strength of the reinforcement.

The interaction of CNTs with the reinforcement by grafting technique is a suitable technique to induce even distribution of CNTs in the composite materials. According to the study this technique can be used prior to the composite development (i.e. Vacuum bagging, Vacuum Resin Infusion etc.). The temperature requirement is low as compared to the chemical vapour deposition method. The dispersion of CNTs also takes place in an organic solvent which has an advantage over the CNTs dispersion in matrix.

To develop samples of CNTs reinforced composite, flat surface mould is selected. Epoxy resin as matrix material while E-glass fibres and carbon fibres are used as reinforcement material. Three layered composites were prepared with four different combinations. 20 samples were produced, utilizing vacuum bagging technique, of dimension (0.40 x 0.30) 1.2 m^2. 16 out of 20 samples are simple composites without grafting of CNTs while others are CNT grafted. Reinforcement is used in the form of woven fabrics. CNTs functionalized with carboxylic group were dispersed in dimethylformamide/PAN suspension through ultra-sonication and sprayed onto the reinforcements in four different volume combinations for each side of the reinforcement i.e. 5 ml, 7.5 ml, 10 ml, 12.5 ml. The process is followed by thermal treatment at 250^{o}C for 3 hours. Reinforcement material is laid on a plain mould after applying lubricant on the base. Resin was applied through hand layup and composites were manufactured by vacuum bagging technique. Mechanical analysis was carried out. ASTM D-3039 standard procedure was followed for tensile tests while ISO 14125 standard procedure was followed for the bending tests of composite.

Their comparison of the results for the tensile, flexural and impact tests showed interesting improvements in the strength of some composite materials. There was no significant improvement in the tensile properties of the composite materials except for

the [CCC] composites grafted with 15 ml of CNT/PAN. Significant improvement is observed in the flexural properties of the composite materials grafted with CNTs.

In order to compare the MWCNTs grafted composites with the Matrix dispersed MWCNTs composites, 4 more specimens were prepared with the same mean quantity of the MWCNTs in CNTs grafted composites. In this case also, the results showed higher raise in the flexural strength up to 70% of the composites but the tensile strength remain unchanged.

The impact strength results also showed valuable information about the usage of the MWCNTs in the composite system. The impact response was improved as the CNT content increased within both the techniques. 15ml – 20 ml of CNT/PAN solution enhanced the impact response to a higher extent whereas the dispersion technique also helped in improving the impact behaviour of the composite material.

This practical study about the effect of carbon nanotubes concludes the influence of techniques by which CNTs are introduced into the composite material and their compatibility with the reinforcement in each technique. The method which is usually carried out to incorporate the CNTs in the composite material is through chemical vapour deposition which consists of very high temperature treatment up to 800 ^{o}C. Since this method requires high temperature conditions which is economically not good and the reinforcements can also be degraded when treated with higher temperatures for longer times, the other methods can play an important role in utilizing the maximum performance properties of the reinforcements. Two methods which are used in this study consists of the grafting method and the dispersion method.

Grafting method deals with the interaction of CNTs directly with the reinforcements to incorporate chemical bonds. In order to increase affinity of CNTs towards the reinforcement to produce chemical bonding, the CNTs are functionalized with the acid group (–COOH) prior to the introduction with the reinforcements. Functionalized CNTs and polyacrylonitrile fibres are dispersed in dimethylformamide through ultrasonic processor so that the aggregates could be properly dispersed in the solvent. The dispersion is sprayed on both sides of the reinforcements followed by the oven treatment at 250 ^{o}C for 3 hours so that bonding could take place. In this study 4 different volumes of dispersion with same concentration of CNTs were used to observe the effects on mechanical properties of the composite materials. After the oven treatment, composites were manufactured using the hand layup process for resin spreading and excessive resin was extracted out using the vacuum bagging.

The grafting technique was expected to give improved tensile properties to the composites because the direct chemical bonding of CNTs with the reinforcements strengthens the branched networks ultimately empowering the material to hold bigger loads with breaking. The results came out to be good for the [CCC] composites but the degradation was more in other combinations. Further to that the higher volumes of CNT dispersion also drastically degraded the tensile properties of the material which could be the reason of slipping layers over each other. The flexural and impact properties of the materials showed particular improvements in 15ml and 20ml of the spray volumes which was quite significant.

Second methodology which is used in this study for incorporating CNTs in the composite materials is by dispersion method that takes place by dispersing the CNTs in the resin through ultrasonication. The resin dispersed with CNTs is then hand laid up on the reinforcement followed by vacuum bagging to make composites.

Ultrasonic dispersion allowed the CNTs to stack themselves in the matrix in such a way that could produce continuous networking structure which reduces the tendency of micro-cracking in the composites. In the experiments, this methodology showed a little improvements in the tensile direction also but the major improvements were seen in the flexural and impact properties. The impact response of the composite was drastically improved that allowed to absorb more force without cracking.

The usage of glass fabric in hybrid composites also influences the flexural and impact properties of the entire material since it has high flexural properties. Combination of glass fabric along with the CNTs network helps in creating a resultant material with significant improvement in flexural and impact strength properties. The reason lies in the continuous stacking arrangement of the CNTs in the matrix. The stacking arrangement of CNTs depends on the diameter of CNTs which makes it able to acquire more surface area towards the matrix. Smaller diameter nanotubes are considered as fine which allows much closely stacking arrangement causing the continuous network of nanostructures which increases the strength of the composite material.

The alignment of stacking is also considered a very important characteristic. Properties along the axis of the nanotubes are enhanced whereas the properties which are dependent on perpendicular alignment are sacrificed. Proper dispersion of the CNTs in the solvent or resin defines the stacking of nanotubes and reduce the agglomeration of the nanoparticles which cause the deficiency in the strength. The higher quantities of the CNTs in the composite also increases the probability of agglomeration which causes the concentrated stress of very smaller areas which leads to ultimate damage.

KARBON NANOTÜP KULLANIMININ KARBON/CAM HİBRİT KOMPOZİTLER ÜZERİNDEKİ ETKİSİNİN İNCELENMESİ

ÖZET

Kompoziler, takviye elemanı ile reçine özellliklerinin duruma uygun olarak belirlenebilmesi ve bu şekilde mevcut yapı malzemeleri yerini alabilecek yüksek performanslı malzeme yapılabilme kabiliyetleri dolayısıyla, her geçen gün daha da yoğun bir ilgi çekmektedir. Çok düşük ağırlıklarda yüksek performanslı kompozitlerin geliştirilmesi konusu araştırmacıların ilgisini gün geçtikçe arttırmaktadır. Onlarca yıl sonra, Karbon Nanotüplerin (KNT) keşfedilmesiyle birlikte araştırmacıların daha yüksek performanslı kompozit malzemeleri yapabilmeleri de ortaya çıkmıştır. Bu buluş, kompozit malzemlerin performaslarında pozitif bir etki de yaratmıştır. KNT lerin genelde malzemelerin mukavemetinde bir artış sağladığı bilinmektedir. KNT lerin kompozitlere katılması için farklı yöntemler bulunmakla birlikte malzeme maliyetleri ise yüksektir. Bu yöntemlerden Kimyasal Buhar Çökertme yöntemi KNT lerin takviye elemanlarına katılmasında çok etkili olmakla birlikte yüksek sıcaklıklara ve uzun sürelere ihtiyaç vardır. Ayrıca yüksek sıcaklıklara çıkılması takviye elemanının mukavemetine de etki etmektedir.

KNT lerin takviye malzemesine graftlama tekniği ile katılması ise; KNT lerin kompozit malzemelerde düzgün bir şekilde dağıtılması için uygun bir yöntemdir. Çalışmalara göre, bu yöntem kompozitlerin üretiminden önce uygulanmalıdır (diğer deyişle vakumla presleme, vakum reçine infüzyon vb. öncesinde). Aynı zamanda bu yöntemde kimyasal buhar çökertme yöntemine göre sıcaklık da düşük tutulabilir. KNT ler önce bir organic çözücü de disperse edildikleri için KNT lerin doğrudan reçine içinde disperslerine göre de avantaj sağlanmaktadır. Bu sebeplerden dolayı çalışmanın ilk ayağında KNT lerin kompozit malzemeye uygulanmazı için graftlama tekniği seçilmiş ve mazlemeler bu yöntemle üretilmiştir.

KNT takviyeli kompozit üretiminde öncelikle düz bir kalıp seçilmiştir. Reçine olarak epoksi malzemesi, takviye olarak da E-cam ve karbon elyaf kullanılmıştır. Dört farklı kombinasyonda üç katlı lamine kompozit malzemeler üretilmiştir. Çalışma boyunca vakumla kalıplama tekniği ile (0.40 x 0.30) 1.2 m2 boyutlarında 20 adet numune üretilmiş olup bu numunelerden 16 sında graftlama tekniği ile KNT ler takviye elemanına dağıtılmıştır. Takviye elemanı ise dokuma kumaş olarak kullanılmıştır. KNT ler önce ultrasonic karıştırıcı vasıtasıyla dimetilformamid/PAN karışımı içinde çözülmüş ve daha sonra sırasıyla dört farklı konsantrasyon (5 ml, 7.5 ml, 10 ml ve 12.5 ml) ile kumaşların her iki tarafında da spreyle püskürtülmüştür. Kumaşlar önce düz bir kalıbın altına kalıp ayırıcı sürüldükten sonra katmanlar halinde serilmiş ve akabinde elle reçine döküldükten sonra ise vakum kalıplama tekniğiyle kompozit malzeme üretilmiştir. Mekanik analiz olarak da ASTM D-3039 standardına göre kompozitlere mukavemet testi ve ISO 14125 standardına göre ise eğilme testi uygulanmıştır.

Malzemelerin mukavemet, eğilme ve darbe testlerinin sonuçları karşılaştırıldığında bazı kompozit malzeme mukavemetlerinde kayda değer iyileşmeler olduğu görülmüştür. 15 ml KNT/PAN ile graftlanmış olan [CCC] kompozitlerin dışında diğer malzemelerde ciddi bir mukavemet artışı bulunmamaktadır. KNT ile graftlanmış kompozit malzemelerin eğilme özelliklerinde ise kaydadeğer iyileşmeler görülmüştür.

Graftlama tekniği sonucunda elde edilen bulgular dahilinde en iyi mekanik etkilerin görüldüğü kompozit malzeme tipi seçilerek, çalışmanın ikinci bir ayağında ise bu tip malzeme üretimi için diğer bir üretim tekniği olarak KNT ler matris içinde dağıtılarak kompozit malzemeye aktarılmıştır. Bu kapsamda; ÇDKNT (çok duvarlı karbon nanotüp) graftlanmış kompozitler ile matris içinde dağıtılmış ÇDKNT kompozitleri kıyaslamak içinde ayrıyeten KNT graftlanmış kompozitlerde kullanılan ÇDKNT miktarı aynı tutularak 4 farklı kompozit malzeme daha üretilmiştir.Bu durumda da, eğilme mukavemetlerinde %70 e varan iyileşmeler görülmüş olup, mukavemet değerleri ise aynı kalmıştır.

Darbe mukavemeti (Serbest ağırlık yöntemi ile-drop weight test) değerleri incelendiğinde ise kompozit malzemelerde ÇDKNT kullanımının etkileri hakkında önemli bulgulara rastlanmıştır. Her iki üretim tekniğiyle (graftlama ve matris içinde dağıtma) üretilen kompozit malzemelerde de KNT uygulamasının artmasıyla darbe mukavemetinin arttığı görülmüştür. 15 ml – 20 ml KNT/PAN solüsyonunun darbe mukavemetini çok arttırdığı görülmekle birlikte matris içinde dağıtma tekniğinin de darbe mukavemetini iyileştirdiği açıkça anlaşılmaktadır.

Karbon nanotüplerin etkileri üzerine yapılan bu pratik çalışmada kompozit malzemeye KNT nin farklı yöntemlerle uygulanması araştırılmıştır. Genelde KNT lerin kompozit malzemeler uygulanması kimyasal buhar çökertmesi ile yapılmakla birlikte, bu yöntemin uygulanması için gerekli olan 800 ^{o} C civarı çok yüksek işlem sıcaklığına ihtiyaç duyulmaktadır. Bu kadar yüksek sıcaklık ekonomik olarak çok verimli olmamakla birlikte, uzun süreli yüksek sıcaklık takviye malzemesine de zarar verebilmektedir. Bu çalışma kapsamında kullanılan iki farklı yöntem olan graftlama ve matris içinde dağıtma yönteminin de kompozit malzemelere KNT ün uygulanması konusunda bir alternatif olacağı da görülmüştür.

Sonuç olarak;

Kompozit malzemelerin üretimlerinde -ağırlığı arttırmadan- matris/takviye malzemesinin arayüzey bağlarınının arttırılması ve dolayısıyla malzemenin de mekanik özelliklerinin iyileştirmenin bir alternatifi ise Karbon Nano Tüplerinin kompozit malzeme içine uygulanmasıdır.

Bu çalışmada, Karbon Nano Tüplerin iki farklı yöntemle (graftlama ve matris içinde dağıtma) kompozit malzemelere başarıyla uygulanabildiği görülmüştür.

Karbon Nano Tüp uygulaması, kompozit malzeme üretimlerine fazladan bir maliyet getirmekle birlikte ticari uygulamalarda ürün maliyetini düşürmek maksadıyla – istenildiği takdirde – graftlama yöntemi ile malzemelere kısmi uygulamalarda yapılabilir. Bu şekilde kompozit malzemeye yük gelecek bölgelerin kuvvetlendirilmesi, eğilme mukavemetlerinde arış veya darbe mukavemetinde iyileşmeler sağlanabilir.

Karbon Nano Tüplerin çok farklı tipleri ve ayrıca boyutları bulunmaktadır. Bu çalışma kapsamında sınırlı sürede ve kaynakta belli bir tip ÇDKNT ile çalışma imkanı olmuş

olmakla birlikte, gelecek çalışmalarda farklı tip ve boyutlarda ÇDKNT kullanımı ile çok daha yüksek mekanik özellikler elde etmek mümkün olabilir.

ÇDKNT lerin ayrıca çok iletken malzemeler olduğu bilinmektedir dolayısıyla mekanik etkiler dışında malzemelere elektriksel özellikler de kazandırılabilir. Bu malzemelerin gelecekte elektrik uygulamalarında da önü açık görünmektedir. KNT takviyeli sistemlerin elektriksel özellikleri gelecek dönemlerde özel kompozit malzemelerin üretilmesi için bir imkan olabilir.

Bilhassa düşük ağırlıkta ve fakat yüksek mukavemetli karbon kompozitlerin KNT ile mukavemetin daha da arttırılması uzay ve yapı uygulamaları için yararlı olabilir.

Mukavemet artışı dışında eğilme özelliklerinde görülen iyileşmeler de, bu malzemelerin otomotiv sanayinde ve spor malzemeleri sektöründe yer bulabileceğini göstermektedir. Eğilme mukavemetinin bilhassa önemli olduğu kasklar, tamponlar, koruyucu padlar gibi malzemeler için KNT ile takviyelendirilmiş kompozit malzemeler kullanılabilir.

Kompozit malzemelerin hibritleşmesinin farklı etkiler gösterdiği görülmüştür. Hibritleşme için malzemenin kat sayısı ve takviye elemanın farklı yönlerde yerleştirilmesi de önemlidir. Gelecek çalışmalarda bu parametrelerin de arttırılması hibritleşmenin etkilerini daha da iyi ortaya çıkaracaktır.

Bu çalışma neticesinde, son ürün özelliklerine göre takviye malzemesi, matris malzemesi ve karbon nano tüp tipi ile boyutunun birbirine uyumlu ve iyi şekilde seçildiği ve ayrıca uygun bir yöntemle üretim yapıldığı takdirde kompozit malzemelerin mekanik özelliklerinde iyileştirme sağlayabileceği görülmüştür.

1. INTRODUCTION

Composite materials are made of at least two different materials. It is superior and unique in performance. Egyptians and Mesopotamian settlers were the first to make use of composites. They used mixture of mud with straws to create strong and durable buildings in 1500 B.C. Mongols developed reinforced bow by the combination of wood, bone and animal glue and then wrapped the bows in birch bark in 1200 A.C. Before the invention of gunpowder these bows were the most powerful weapon on earth. Modern era of composites starts with the invention of plastic in early 1900 that replaced natural glues and resins. Plastics are not strong enough to provide support for structural applications. Reinforcements were needed, that can provide rigidity and strength. In 1935, high performance fibres like glass fibre were developed. Plastic was light and glass fibre was strong; their combination introduced a new trend in material sciences. In 2nd world war alternative light weight but high strength materials were needed for applications like military aircraft industry. Fibre glass composites were also used to shelter the electronic radar systems as they are transparent to radio frequencies. Until the utilization of drop-weight testing machines in 1970's to estimate the impact strength, the dynamic repose of composites was not focused (Lifshitz, 1976).

After the world war 2nd, composites industry started growing in full swing. The introduction of surf board made of glass fibre composite revolutionized the sports industry. Composites are now involved almost in every sport equipment e.g. bats, rackets, wickets, baskets, sticks etc. In peace days, lower demand of military products, scientist found the application of composites in other fields of life. First commercial composite boat hull was developed in 1946. Brandt Goldsworthy, developed light weight composite surfboard which revolutionized the sports industry. New manufacturing techniques were developed with the passage of time driven by need for alternative materials. In 1970's the composite industry began to mature. Better resins and advanced reinforcement fibres were developed that found applications in civil, transport, aerospace, agriculture, medical and sports.

For last 30 years composites are the most important industrial materials. Composite industry is still evolving and pushing the limits to acquire advance materials, designs and manufacturing techniques (Abdullah). A detailed study of production and evaluation of properties of Carbon Nanotubes reinforced hybrid composites has been carried out in this project, focusing the effect changing quantities of CNTs in different combinations. For this purpose Vacuum bagging technique is chosen as a processing route for the polymer matrix composites.

Composites have been used in the marine industry since the early 1950's. Composite vessels feature in almost every area of the marine market, from small leisure craft to large yachts, fishing boats, lifeboats, passenger ferries, pilot boats, floating shopping centre, hulls of catamarans, hovercrafts, sail and surf boards, dry dock caissons and sluice gates. The appearance of composite vessels is aesthetically pleasant and is weather and corrosion resistant, resulting in reduced maintenance compared with other metallic materials. High performance resins are combined with high strength fibres such as glass, carbon and poly aramid to manufacture vessels with outstanding physical properties. Specialised resins impart toughness to conventional polyesters; have enabled the production of high performance craft which can withstand extreme impact and flexural loading without cracking during extreme performance. Developments in other products such as core materials, now allow the manufacture of light weight, stiff, high performance craft that are extremely resistant to the marine environment.

1.1. Purpose of Thesis

The purpose of the this thesis is to manufacture the CNTs reinforced Carbon, Glass and Hybrid composites through grafting technique, and to study the effect of CNTs on the mechanical properties of the composites. Furthermore to investigate the optimal amount of CNTs to get the improved mechanical results in various composites.

1.2. Literature Review

Materials are combined in a way to minimize the effect of their deficiencies in a composite that enable us to make better use of their properties. This process freed the designer of constraints related to the selection of material for a specific use. Different

phased materials are combined to give a product with optimized properties called Composite material. Tougher and lighter materials have made possible to fulfil particular design requirements. Individual components remain separate and distinct in the composite structure (Chapman & Hall, 1998). Composites materials are among the oldest and newest of structural materials. Composites are developed by mixing at least two materials to produce a non-reversible material. They consist a bulk material called Matrix and a reinforcement of some kind and are characterized by high strength, better stiffness, cost, high performance, electrical properties and simple manufacturing techniques (Chapman & Hall, 1998) (Awan, et al., 2009).

Main function of the matrix is to surround the disperse phase i.e. reinforcement. It keeps the fibres together and oriented. It also works as a medium that distribute external load over the fibres to minimize the effect of load. Factors that can affect the mechanical properties of a composite are fibre type, fibre volume fraction, orientation of the fibres and the thickness of the laminate. Mechanical characteristics of constituents with respect to composite are shown in figure 1.1 (Robertson, et al., 1992).

After the Iijima's report about CNTs in 1991, scientists have been taking more interest in the unique atomic structure and properties of CNT's, such as high aspect ratio, high strength-to-weight ratio, good mechanical properties (their axial tensile strength and elastic modulus were theoretically expected to be as high as 200 GPa and 1–2 TPa (Robertson, et al., 1992), respectively), extraordinary electrical and thermal properties (electric-current-carrying capacity as compared with copper wires is 1000 times higher (Avouris & Collins, 2000), thermally stability retains up to 2800°C in a vacuum or inert atmosphere and thermal conductivity is about twice to that of diamond (Kim, et al., 2001)). These extraordinary properties of CNTs entitles themselves with new scientific and technological opportunities as unique ideal filler material in composites.

Composite manufacturing process is a goal oriented process. Each process has its own characteristics that define the type of product to be produced. It allows the composite industry to provide the best solution to the customer need. It involves various operations depending on the availability of technology, provided facilities and personnel skills. The variation in the manufacturing process is also due to vast variety of composite materials and their applications. End use, production volume, size of product, economic targets, labour intensity, materials involved, required skills, surface complexity and appearance, production rate, tools and required equipment are the

factors that are considered while choosing a manufacturing process to produce a composite with desired specification. A consistent product can be achieved by controlling parameters like fibre thickness, resin type, fibre volume fraction and its orientation in the resin. It follows steps to minimize cost, reduce voids and internal stresses (Scott Bader Company Ltd, 2005).

To achieve the manufacturing goals different combination of tools, materials and processing methods are combined to developed manufacturing technique. It is evident that the technique for manufacturing composite is based on the complexity of product and also batch size.

Resin alone is very brittle and its excess can weaken the product. As the vacuum bagging involves hand layup hence the laminate will always begin in oversaturated state and the process still depends on factors like reinforcement, resin type and time. Vacuum bagging utilizes the technique of drawing excess resin out of the reinforcements through vacuum pressure. This technique results in the minimum amount of resin holding the reinforcement and eventually the laminate approach prepreg levels. Figure 1.1 shows the vacuum bagging arrangement and stepwise introduction of resin into reinforcement by vacuum pressure. Vacuum bagging is a little clean process, splashing, spattering and hovering can be avoided. It provides an average working environment. However it is still important to ventilate the work place and follow appropriate safety measures (Ratna, 2009).

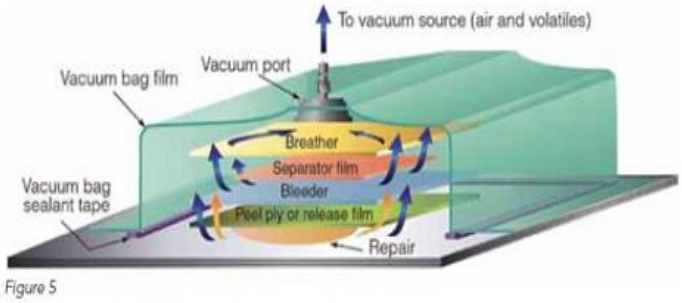

Figure 1.1: Vacuum Bagging Set-Up.

95% of the fibres used in reinforced composites are the glass fibres. They are cheap, easy to manufacture and gives high strength and stiffness to the composite. It has lower density with higher resistance to chemicals and good insulation properties but can break easily if prone to prolonged tensile stresses. However it shows good tensile strength if higher stresses are applied in shorter time frames.

Molten glass is drawn into fine threads to produce glass fibres. Freshly drawn glass fibres are protected from contact with hard surface and atmosphere to get undamaged and defect free fibres. Glass fibres are inorganic and strong fibres but lack rigidity due to their molecular structure. S-glass (higher strength glass) has better properties than E-glass in terms of strength and thermal stability but its use is limited due to higher cost (Chiu & Cheng, 2002). Glass fibres are available in short, long and continuous form. The dimension of fibres is characterized by aspect ratio. Longer fibres with smaller diameter add more strength to the composites. Ends of the fibre carry fewer loads with respect to its diameter. Surface imperfections causes fracture in the glass fibre (Starr, 1995).

Bonding at interface between fibre and matrix is important factor in the determination of mechanical properties of fibre reinforced polymers. It is noticed that the adhesion between the resin matrix and fibre is the main precondition for the transfer of load. Laminates are subjected to various predominated stresses on the basis of their end use. The properties and fracture analysis of polyester panels is a complex and demanding field of study. Predominant load is considered as basis for the selection of composite for an application (Bunsell, 1988).

Sandwich structure composites are special category of composite family. They are fabricated by attaching two thin, strong and stiff skins to light weight core material. Separating two materials with a light weight material in between increases the stiffness and strength of the structure at a low weight and cost. It enables light weight construction, freedom of design and low cost alternative to many counterparts (Paolo, et al., 1997).

The quality of the composite and its reproducibility depends on the route of production. Improper fibre to resin ratio, unoriented, broken fibres and non-uniform fibre distribution that results in matrix rich regions, gaps, overlaps or other faults in the arrangement of reinforcement layers, air voids, poorly bonded interlaminar regions, incorrect state of resin cure, resin cracks, transverse ply cracks due to thermal mismatch stresses, localized damage due to mechanical impact around machined holes are the defects may be present in any manufactured composites (Jeong, 1997).

Various procedures are standardized to evaluate the defects in any manufactured composite. Standards are used to characterize the properties of the composite.

1.2.1. Polymer Matrix Composites (PMCS)

Materials like polyester and epoxy have limited use on their own for structural application because their mechanical properties are not high with respect conventional construction materials like metals. One good aspect of these materials is that they can be easily formed into complex shapes. If reinforced with some strong material can improve mechanical properties. Similarly high performance fibres have high tensile and compressive strength but practically these properties cannot be utilized due to the fact that surface damages are produced when stressed. In order to overcome this problem high strength fibres are combined with resin to produce composite material having optimized properties of all the constituents. By the combination of different resins to reinforcements, composites with good tensile, bending, toughness properties along with good resistance to environmental degradation can be achieved. Figure 1.2 shows the fibres surrounded by matrix in composite structure (Roylance, March 24, 2000).

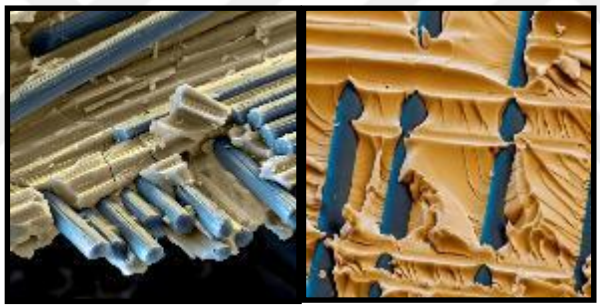

Figure 1.2: Matrix surrounding fibers.

Polymers possess an important property that they are composed of long chain molecules which has many repeating units. Their use is very common because they infiltrates very easily. According to the response to heat treatment, they can be classified as Thermosets and Thermoplastics.

Thermosets are branched chains polymers. Once cured cannot become liquid again on heating. The resin and hardener or resin and catalyst are mixed, a non-reversible reaction starts giving a hard infusible product with 3D bonded network of polymer chains. Phenolic resins produce volatile by products while others don't. Above glass transition temperature the mechanical properties depletes because thermosets become flexible. Due to lower viscosity they infiltrate very easily and contribute to the strength and stiffness of the product. Thermosets show good resistance to chemicals. They are

brittle laminates with limited high temperature sustainability especially in hot-wet conditions. Thermosets wets the reinforcement more easily than thermoplastics (Pickering, 2006).

1.2.2. Epoxy resin

Epoxies are being used in order to bind the strengthening fibre and locking them in a position so that they can sustain their position to do their job. Fibres are placed in specific directions in the fibre reinforced structures to focus the reinforcement according to need and the epoxy helps in retaining the position of fibers where they are needed. The aim of the epoxy matrix is to surround the fiber and transfer the loads to the fibers even though it is a strong material itself. It helps in protecting the fibers from damage and offers impact resistance (Christine, n.d.).

Thermosetting polymer resins in which the resin molecule contains one or more epoxide groups are known as Epoxies. The chemistry can be customized by the molecular weight or viscosity as required by the end use to make it a perfect option. Two main types of epoxies (figure 1.3), glycidyl epoxy and non-glycidyl are available. Glycidyl epoxy resins can be briefly defined as either glycidyl-amine, glycidyl-ester, or glycidyl-ether. Non-gylcidyl epoxy resins are either cycloaliphatic or aliphatic resins.

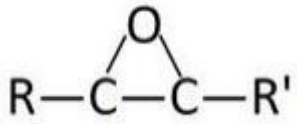

Figure 1.3: Basic Epoxy Structure.

One of the most common glycidyl epoxy resins is manufactured by using Bisphenol-A, and is synthesized in a reaction with epicholrohydrin as shown in figure 1.4 (Fink, 2013). The other commonly used type of epoxy is novolac based epoxy resin. These resins have a thermosetting nature and they have a requirement of suitable hardener to let them cure.

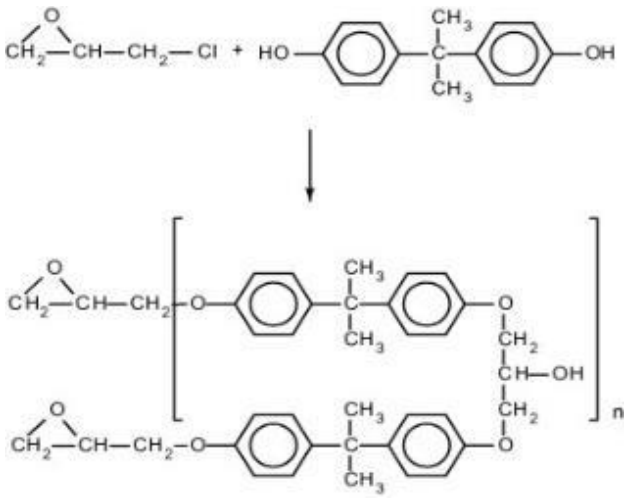

Figure 1.4: Synthesis of an epoxide oligomer.

Epoxy resins require a curing agent for the setting up which is commonly called a hardener. Usually amine based curing agent are used generally. In polyester or vinyl ester resins the resin is catalysed with a small (up to 3%) addition of a catalyst, whereas epoxy resins usually require the addition of the curing agent at a much higher ratio of resin to hardener.

Although being very resistant cured epoxy degrades on exposure to UV. The surface attains a chalky appearance and loses strength. A UV protection layer can be beneficial in this respect. At elevated temperatures Epoxy loses strength.

Epoxies have influenced a wide range of applications such as fiber-reinforced plastic materials, general purpose adhesives and strong chemically resistant coatings and finishes.

1.2.3. Reinforcement material

Reinforcement adds mechanical properties to the composites. Primary function of reinforcement is to carry load along the direction of their orientation. Mechanical properties of the fibre, its surface interaction with resin, orientation and fibre volume fraction are the factors that define the contribution of reinforcement in composites and most of its properties. For higher end use synthetic reinforcements are used but natural reinforcements are also for many applications (Taranu, 2008). Carbon Nanotubes are also proven to be a source of extraordinary value addition to the reinforcements, such

as Glass and Carbon, as they share the high tensile properties to the resultant reinforcement.

1.2.4. Natural reinforcements

Natural fibres, Asbestos (Mineral Fibres), Cellulose (Vegetal Fibre), Collagen and Silk (Animal Fibre) are used as reinforcement for many application. Natural fibres are cheaper source of reinforcement but have lesser mechanical properties than manmade fibre (Taranu, 2008). Wood Plastic Composites (WPCs) have acquired a wide range of applications in construction and building sector, usually as lumber for railing and decking systems. They also have an advantage of higher stiffness than thermoplastics and are equipped with a natural "wood" look (Mohd Ishak & Taib, 2015).

Changing trends in the world and sustainability issues have forced the manufacturers to use more environment friendly materials such as biodegradable composites. The preparation of the biodegradable composites involves combining of biodegradable polymers with natural fibres (Mohd Ishak & Taib, 2015). Polylactic acid (PLA) can be served as biodegradable polymer and kenaf fibre (KF) as in natural fibre or filler. For sustainable economic development, bamboo industry has been mentioned as an effective source for bio composite fibres, which offers minimal production cost and can bring a revolution in the world of supply chain and manufacturing (Abdul Khalil, et al., 2015).

Synthetic fibre-reinforced composites having good physical properties with high performance at low weight can be substituted by jute-reinforced thermoplastic laminates and composites. Jute fibre composites have sufficient potential to influence the automobile industry, the footwear industry, construction, furniture, and the toy sectors (Khan & Khan, 2015).

1.2.5. Synthetic reinforcements

1.2.5.1. Aramid fibers (Kevlar)

Aramid fibres are man-made organic fibres based on aromatic polyamide chains. DuPont developed aramid fibres. They are closely related to nylon. Aramids have low solubility due to aromatic groups they cannot be drawn so are spun from liquid crystalline solution. They have pale yellow color with high strength and low density hence gives very high specific strength. Aramids have fair compressive strength i.e.

1/8th of its tensile strength because of anisotropic structure. They have good impact resistance with elongation to fracture value (3.5-4%) in Kevlar 29 and Compressive strength of aramid fibres is similar to E-Glass. In longitudinal direction it is very difficult to stretch the aramid fibre because of its structure but in other directions the planes are loosely attached. They show good resistance to abrasion, chemical and thermal degradation. Prolong exposure to UV light can cause degradation to Aramid fibres (Chatzi & Koenig, 1987).

1.2.5.2. Carbon fiber

Polyacrylonitrile (PAN) based fibres having unusual properties of graphite and is obtained by controlled pyrolysis of an organic fibre "PAN" precursor in 4 steps (Transformation, stabilization, carbonization and graphitization). Its Degree of molecular chains alignment depends on processing route and Planes are oriented along the fibre axis. Its main feature is alignment of graphite sheets along fibre direction. Commercial Carbon fibre is normally sized with low molecular weight epoxies to avoid defects due to rubbing among fibres, easy handling and improve bonding with resin. Carbon fibres show anisotropic behaviour and its mechanical properties depend on amount of graphite and basal planes. Strength of carbon fibre depends on percentage of defects. (Inclusion voids). It has good thermal and electrical properties and negative CTE in fibre direction (Department of defense, USA, 23 Jan 1997).

1.2.5.3. Glass fiber

Glass Fibre is SiO_2 based amorphous fibre. It contains 50-60% of SiO_2 with host of other metal oxides. Its raw material is cheap but the processing route is not sophisticated. It is the backbone of Information Technology Sector. The properties of Glass Fibre can be tailored by altering its chemical composition. They show isotropic behaviour. Glass fibres have high strength but stiffness. Mechanical properties decay above 400-500^{o}C and ultimately melts. Glass fibres corrode in alkaline environment and are not resistant to water especially sea water but are not expensive with respect to counterparts (Chapman & Hall, 1998) (Department of defense, USA, 23 Jan 1997).

S-Glass has high tensile strength and modulus with better wet strength retention than E-Glass and is used in aerospace and ballistic armour. Its composition is 65% SiO_2, 25% Al_2O_3, and 10% MgO. Mixture of raw oxides is directly melted and is drawn in

melted form through electrically heated platinum bushes. Commercial glass fibre is normally sized with low molecular weight epoxies to avoid defects due to rubbing among fibres, chemical attack, easy handling and improve bonding with resin (Kinsella, et al.).

The Glass should be smooth with no gaps among the strands to get the best surface finish. It is better to use number of layers of lower weight glass instead the heavy and bulky one to fit the contours of the part. The bridging in the corner causes resin race tracking and leads to poor fill. The glass is laid try into the mould. Different methods are used to keep the glass in place especially on vertical surfaces. It can be achieved by using adhesive backed glass or by using adhesive sprays to standard glass fabric. The adhesives are specially developed for resin infusion process. Limitation of Glass fibre is less tolerance to prolonged loading temperature and moisture. Its disadvantage is further pronounced by the fact that the brittleness leads to a catastrophic failure without any prior warning. Mechanical properties of different Glass fibre types are listed in table 1.1 (Lund, 2010).

The initial application of the glass fiber was to protect radar equipment. It was also used as ducting in airplane engine nacelles. The first usage of the fiberglass in the main airframe construction was in Spitfire in England but it didn't come out into production.

In 1953 Chevrolet put their first production of their model Corvette in which the body was composed of fiberglass composite. Fiberglass bodies significantly reduce the weight of the machine but their commercial production hasn't yet succeeded the metallic industry. Instead they are particularly famous for replacement body parts, custom and kit auto markets. The tooling costs are also low as compared with metal press assemblies.

Table 1.1: Mechanical Properties of Different Glass Fibers.

	A-glass	C-glass	D-glass	E-glass	ECR-glass	AR-glass	R-glass	S-2-glass
Density (g/cubic cm)	244	2.52	2.11-2.14	2.58	2.72	2.7	2.54	2.46
Tensile Strength (Mpa) at 196°C		5380		5310	5310			8275
Tensile Strength (Mpa) at 23°C	3310	3310	2415	3445	3445	3241	4135	4890
Tensile Strength (Mpa) at 371°C				2620	2165		2930	4445
Tensile Strength (Mpa) at 538°C				1725	1725		2140	2415
Modulus of Elasticity (GPa) at 23°C	68.9	68.9	51.7	72.3	80.3	73.1	85.3	86.9
Modulus of Elasticity (GPa) at 538°C				81.3	81.3			88.9
Elongation %	4.8	4.8	4.6	4.8	4.8	4.4	4.8	5.7

1.2.5.4. Carbon nanotubes

CNTs have proven to be the ultimate strongest known material which makes the discovery of CNT a benchmark in nanotechnology (Iijima, 1991). CNTs are actually hollow cylinders of carbon atoms. They have an appearance of rolled tubes of graphite, and their walls have hexagonal carbon rings. Two types of nanotubes are there: single-walled nanotubes (SWNTs) and multiwalled nanotubes (MWNTs), which can be

differed by the arrangement of graphene cylinders (Ganesh, March 2013). Figure 1.5 represent SWCNTs and MWCNTs structures.

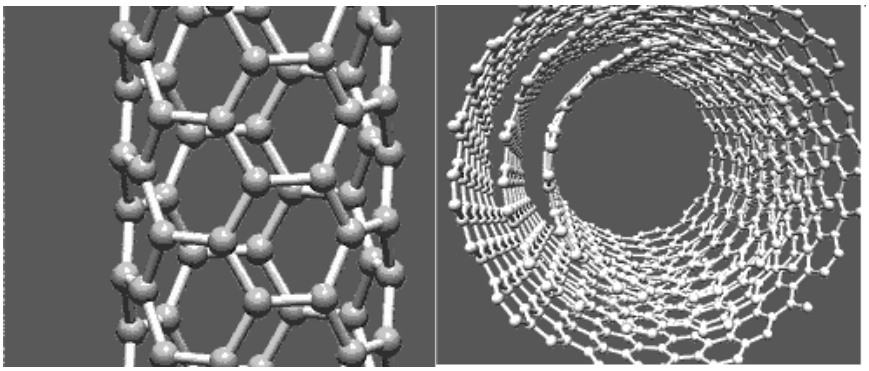

Figure 1.5: Single Walled CNT and Multiwalled CNT.

The composites can be more effectively reinforced by CNTs as compared to conventional fibres as they share extraordinary large interfacial contact area with the matrix but with very less weight penalty. Scientists have observed that addition of only 1% of CNTs by weight in a matrix material can result in the increase in stiffness of the composite film by 36–42% and the tensile strength by 25% (Qian, et al., 2000). Apart from using independently CNTs can be used in combination with other common reinforcements like poly-acrylonitrile fibres, carbon fibres and glass fibres for improving, mechanical, electrical and thermal properties of the composites (Fernándeza, et al., 2013) (Wang & Qiu, 2010).

There are a few problems associated with the use of CNTs which include the preparation of structure controlled CNTs retaining the high purity and consistency leading to high performance and the breaking up of bundled CNTs to uniformly disperse and distribute them within a polymer matrix. One more issue is improvement of load transfer from matrix to CNT reinforcement (Du, et al., 2007).

For Carbon Fabric there have been a lot of studies which are focused on achieving an effective method for CNT/CF hybridization. The results can be summarized in two categories: the direct growth method of CNTs on the CF surface by chemical vapour deposition (CVD) (Kim, et al., 2013) and the spray coating of CNTs on the CF surface (He, et al., 2007) or electrophoresis (Bekyarova, et al., 2007).

It has been observed that the CNT growth technique can achieve much higher concentrations of CNTs in a composite as compared to that of dispersion in the matrix. This technique also avoids common problems with CNTs like agglomeration, filtration

and increased viscosity which puts some hurdles in determining the actual CNT contents (Romanov, et al., 2015). Figure 1.6 shows the CNT growth on carbon fiber through SEM image.

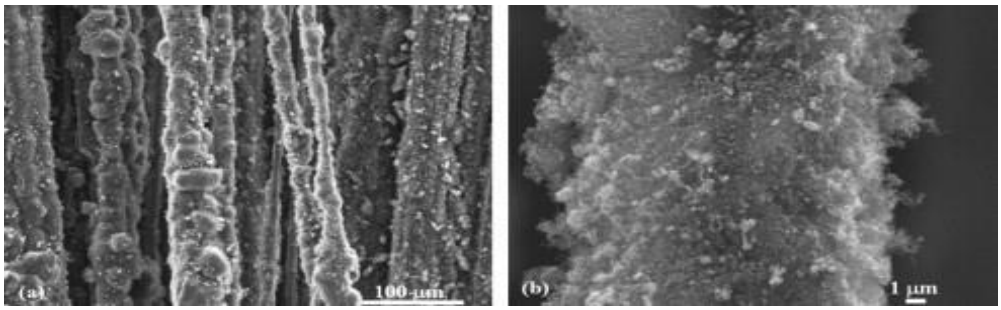

Figure 1.6: SEM image of grown CNTs on carbon fiber (a) a low magnified image of several fibers covered with CNTs, (b) an individual fiber covered with CNTs.

The main problem reported with the CVD-based method is that it requires high-temperature process over 700 C and layers of ceramic buffers to prevent the deactivation of catalysts (Kim, et al., 2012). In addition to that the mechanical properties of CFs are also degraded after the CVD process (Kim, et al., 2013).

Multiwall carbon nanotubes (MWCNTs) are usually preferred over single wall carbon nanotubes (SWCNTs) due to their lower cost, easier production and better dispersion (Sharma & Lakkad, 2015).

Volkan Eskizeybek, Ahmet Avcı & Ahmet Gülce in 2015 reported a decrease of 11% in ultimate tensile strength of glass fabric/CNT/epoxy multi scale composite whereas toughness was increased up to 57% (Eskizeybek, et al., August, 2015). Geunsung Lee, Kwang Duk Ko Young Chang Yu, Jinyong Lee, Woong-Ryeol Yu, Ji Ho Youk in 2015 observed a 22% enhancement in the tensile strength of the CNT/PAN grafted carbon epoxy composite as compared with the pristine CF composite (Lee, et al., 2015). The CNT grafting process through spray coating can be a helpful tool to ensure high mechanical properties in a localized area of a composite material.

Eslam Soliman, Usama Kandil and Mahmoud Reda Taha observed a significant improvement in the flexural strength, modulus and toughness of the off-axis specimens of epoxy nanocomposites reinforced with carbon woven fabric. The addition of 1.5 wt. % of COOH-MWCNTs in epoxy resulted in the average increase of 28% and 19% in flexure strength and flexure modulus respectively (Soliman, et al., 2014).

The grafting of CNTs on the reinforcements is particularly being used for the hybridization of carbon fabrics. MWCNTs functionalized with carboxylic group can be grafted onto the fabric body in order to improve the properties of the reinforcement. Mohammad M. Fares, Fahmi A. Abu Al-Rub, and Khansa'a H. Massadeh produced MWCNTs-g-carbon precursors by using COOH functionalized MWCNTs with PAN which increased the thermal stability up to 850^{o}C and also improved the UV absorption (Fares, et al., 2015). The step by step formation of MWCNTs-g-PAN carbon fibre precursor can be seen from figure 1.7.

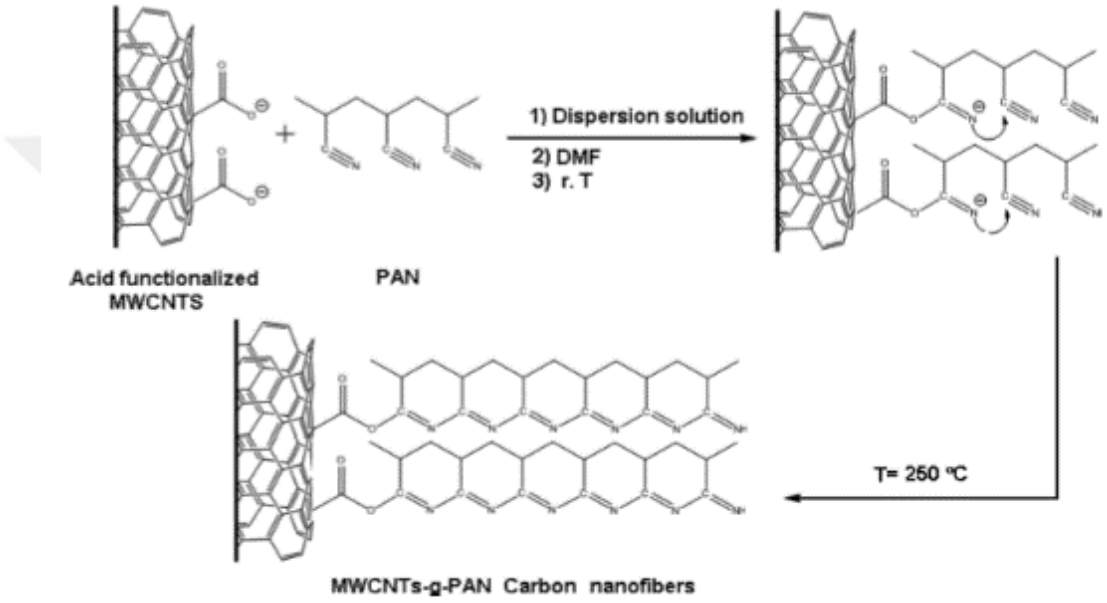

Figure 1.7: Schematic Illustration for the Formation of MWCNTs-g-PAN Carbon Fiber Precursors.

The interaction of MWCNTs with the glass fibre coating was investigated by the researchers to know the possible chemical bonding between the two entities. Fibre coating plays a significant role on the deposition of MWCNTs onto glass fibres and their bonding. The chemical analysis of the glass fibres and its coating indicates the presence of multiple reactive functional groups such as carbonyl, hydroxyl, and epoxy groups that can react with the carboxyl and hydroxyl functional groups MWCNTs as shown in figure 1.8 (Ku-Herrera, et al., 2015).

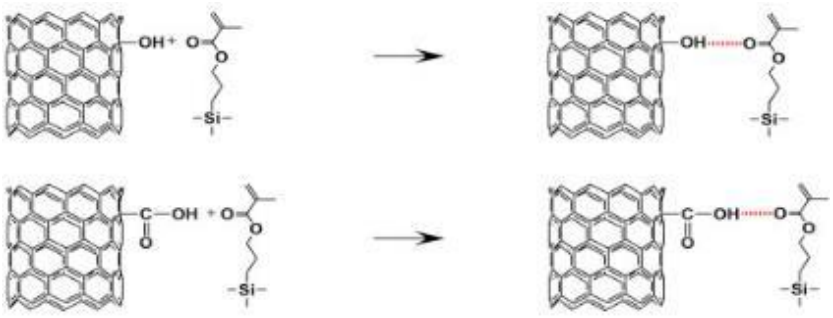

Figure 1.8: Schematic reaction of the functionalized MWCNTs with glass fiber coating.

Proper dispersion followed with adequate interfacial adhesion is required in order to make CNTs as an effective reinforcement in polymer composites (Ma, et al., 2010).

1.2.6. Hybrid Composite Materials

Hybrid composite laminates are those comprising of multiple type of reinforcement materials. These composite materials facilitates the customization of the reinforcement properties by using different types of reinforcements. Carbon and glass fibres have a wide range of application which demands high strength, low weight and low cost. The reinforcements used to make a hybrid material help in improving certain properties, e.g., the glass fibres has a lower modulus with higher strain-to-failure in comparison to carbon fibres. In this case the flexural strength of carbon fibre-reinforced polymer (CFRP) composites can be significantly improved by substituting some of the carbon fibre laminas by glass fibre (Kalantari, et al., 2015).

The resultant composite manufactured by carbon and glass fibre hybridization not only influences the flexural strength and stiffness but other key properties also such as cost and weight. Subsequently the glass fibres are heavier and cheaper than carbon fibres, hybridization of a CFRP composite through the incorporation of glass fibres leads to a lower material cost but higher density. The weight and cost reduction are the two major concerns that leads in the designing process of hybrid composite structures (Kalantari, et al., 2015).

The length of fibre, fibre orientation, and fibre shape and fibre material of the reinforcements are the few influential factors that affect the performance of the composite’s mechanical performance.

1.2.7. Ultrasonication

It can be explained as the process of applying ultrasound energy to agitate nanoparticles in a solution for desired purposes. An ultrasonic bath or an ultrasonic probe is used as Sonicator. This technique is based on the principle that attenuated waves are induced in the liquid molecules when ultrasound propagates through a series of compression. These vibrations tend to overcome the interactions between the CNTs which results in the dispersion.

The sonication treatment provides the ability to the surfactant molecules to be adsorbed onto CNT surface by overcoming the interactions produced by the hydrophobic, electrostatic and van der Waals forces (Yu, et al., 2012). Firstly the liquid is adsorbed on the CNT surface which reduce the size of big bundles to smaller bundles. After sufficient time the solution contains individual CNTs and tiny bundles of CNTs. The main parameters of the sonication process are time and power. The power of the sonication system should be optimal because lower powers cannot separate the CNT bundles and the higher power could create more than required shear stress on CNTs resulting in their damage.

The power is more important characteristic of the sonication system as compared with the sonication time (Yu, et al., 2012).

1.2.8. Tensile test

The concept of tensile test can be explained through normal stress and shear stress. A specimen or sample is gripped at both ends and is pulled apart. The amount of stress in the specimen can be found by calculating the force applied by the grips over the cross sectional area normal to the applied force. The ultimate tensile strength is the highest value of force that the tester measures for a specimen while modulus of elasticity is found as the slope of the line presenting stress as the function of strain (Hermann & Locke , November 2006).

The international standard for tensile testing is given by ASTM and its designation is “D 3039/D 3039M – 00 “. It determines the in plane tensile properties of Polymer matrix composites reinforced by high modulus fibres. Thin flat strip with rectangular cross section is mounted on a mechanical testing machine and a monotonic load is applied in tension while the load is being recorded. The ultimate strength of material

is the maximum load carried before failure. ASTM D- 3039 basically determines the in plane properties of high modulus fibre reinforced polymer composites. The reinforcement forms are limited to continuous or discontinuous fibres while the test specimen of polymer composite is symmetric with respect to test direction (ASTM D 3039/D3039M – 00, , 2002).

When a material is subjected to a load, the force distributes itself through the material in the form of internal stresses. The behaviour of material during failure is determined by the amount of stress that exists inside it. Mathematically, Stress is Force applied over the area normal to the applied force while shear stress is the force applied parallel to the area. Stress is expressed in Pascal (Pa). Material under stress deforms either by stretching, contracting or bending. This resulting deformation is called strain and can be defined as change of length of specimen divided by the total length of specimen. Strain is a dimensionless quantity (Askeland, 1990).

1.2.9. Bending test (Mid Span Loading)

It determines the properties of sandwich constructions that are subjected to flat wise flexure. Load is applied is applied in a manner to produce curvature of the sandwich facing planes. Flexural stiffness, core shear strength and shear modulus or the facing compressive and tensile strengths of sandwich construction can be evaluated. For this purpose ISO standard 14125 is followed. Sandwich stiffness and core shear modulus can be evaluated by calculating deflection of sandwich flexural specimens. ISO 14125 allows the use of either single (midspan) loading or two-point loading. These are generally called three-point and four-point flexure testing, respectively. The configuration of flexure can be altered to perform both the tests. In order to perform three points loading test, one loading head is removed while the remaining one apply load at the centre of the specimen (Standard test method for flexural properties of sandwich constructions.).

1.2.10. Impact test (Drop weight test)

This test methodology is used to determine the damage resistance of composites facing a drop-weight impact event (ASTM International). ASTM D7136 test standard was used to carry this test. This test is carried by a fall of a mass with a semispherical tip from a certain height onto the laminate. The weight of the impactor was 35 kg. This

test was not carried for the standardized specifications since ASTM D7136 specifies a value of 5.50 kg for the impactor, but allows the use of other values. The energy with which the impactor strikes the test specimen is dependent on the mass of impactor and the height from which it is dropped (Justo, et al., 2015).

The area of the damage and the indentation depth helps in measuring the impact resistance of the specimen.

2. EXPERIMENTAL WORK AND METHODOLOGY

2.1. Safety

Standard safety procedures are strictly followed. Disposable gloves, lab coat, safety goggles are plastic shield to surround the tension test apparatus to avoid exploding bits of composite on failure during mechanical testing are practiced for personal safety.

2.2. Preparation of CNT/PAN Dispersion

2.2.1. Materials and equipment for CNT/PAN dispersion

The materials and equipment required for preparing the CNT/PAN dispersion are listed in this section. The ultrasonic mixer was used in a soundproof casing to avoid ultrasonic waves.

- DMF
- PAN
- Multiwalled carbon nanotubes (COOH functionalized)
- Beaker
- Pipette
- Ultrasonic processor (figure 2.1) with sound proof case
- Weighing Balance

Figure 2.1: Ultrasonic processor.

The table 2.1 shows the chemicals and equipment required to prepare the dispersion of CNTs and PAN. Moreover there specifications are also described.

Table 2.1: Materials and equipment for CNT/PAN dispersion.

Material/Equipment	Specification
CNTs	COOH functionalized MWCNTs from Cheap Tubes Inc. 0.3 wt. % of PAN/DMF Solution.
PAN	0.3 wt. % DMF from AKSA Inc.
Dimethylformamide (DMF)	98% Purity / 0.944 g/ml Density from MERCK. Four volumes to be sprayed/reinforcement as listed below: 10 ml / 15 ml / 20 ml / 25 ml
Ultrasonic Mixer	Heilscher UP 400S (400W)
Sonotrode	H3
Sonication Time	60 min in 2 parts

2.2.1.1. MWCNTs specifications

MWCNTs were procured from Cheaptubes Inc. USA which consisted of below mentioned specifications.

Outer Diameter	: 30-50 nm
Length	: 10-20 µm
Purity	: 95 wt. %
-COOH content	: 0.73 wt. %

2.2.2. Procedure for CNT/PAN dispersion

The experiments includes four different volumes of CNT/PAN DMF solution, i.e. 10 ml, 15 ml, 20 ml and 25 ml, that would be sprayed on each reinforcement separately to observe the variation in properties and the optimum amount of CNT/PAN required to achieve the maximum mechanical properties. In order to prepare the solution first of all 0.3 wt. % of PAN suspension was made in DMF followed by the introduction of 0.3 wt. % COOH functionalized CNTs with respect to the PAN suspension. Figure 2.2 shows the setup made for the sonication process. CNTs were dispersed in the suspension by sonication for 60 mins in two parts. It was insured that there are no aggregates of CNTs in the solution.

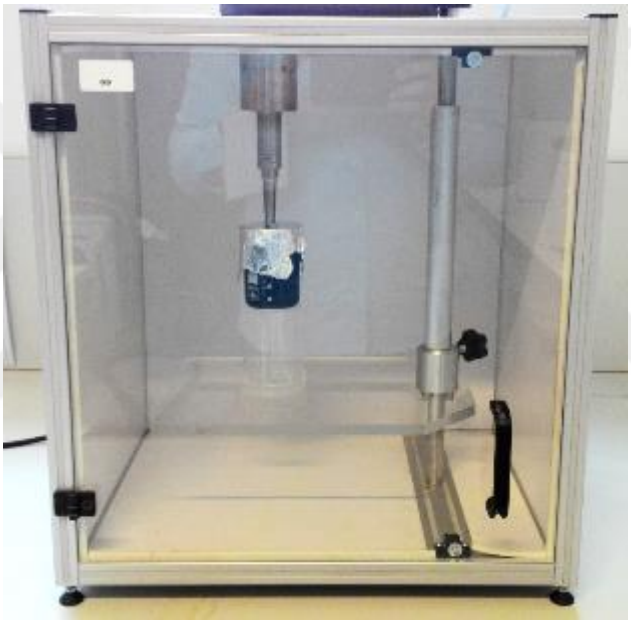

Figure 2.2: CNT dispersion through sonication.

2.3. CNT grafting methodology

2.3.1. Materials and equipment for CNT grafting

The materials and equipment required for preparing the grafted reinforcements are listed in this section. Spray coating technique was carried to incorporate CNTs grafting on reinforcements.

- Spray Gun (figure 2.3)
- Iron Frame (figure 2.4)
- Oven (figure 2.5)

Figure 2.3: Spray gun.

Table 2.2: Materials and equipment for CNT grafting.

Material/Equipment	Specification
Spray Gun	Low Pressure spray gun (0.08 mm nozzle)
Iron Frame	30 x 40 cm Frame with grip to hold the reinforcement during thermal treatment
Oven	Heraeus
Oven Temperature	250 ºC
Oven Time	180 min

Figure 2.4: Clamped reinforcement.

Figure 2.5: Oven.

2.3.2. Procedure for grafting CNTs

Reinforcements were cut according to the frame size and clamped on the iron frames as seen in figure 2.4 to avoid any dimensional instability during the thermal treatment. Required CNT/PAN dispersion was poured into the spray gun for each side of the reinforcement separately. CNT/PAN dispersion was sprayed on the reinforcements slowly and with constant speed to ensure maximum homogeneity in the spreading of the CNT/PAN grafts. The dispersion was sprayed in 4 different volumes followed by the oven treatment for 3 hours at 250^{o}C. Figure 2.6 and 2.7 shows the prior and later to spray pictures of carbon and glass fabrics.

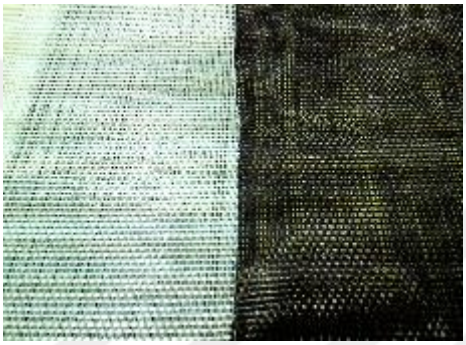

Figure 2.6: Without CNTs and with CNTs glass fabric.

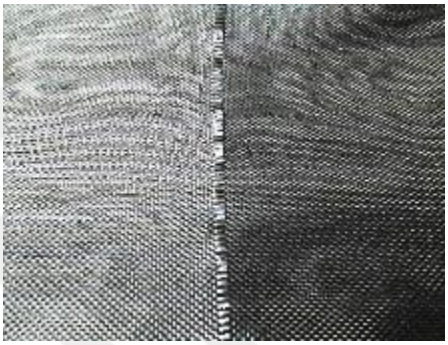

Figure 2.7: Without CNTs and with CNTs carbon fabric.

2.4. CNT-Epoxy dispersion (alternative technique)

After carrying the experiments for the grafting technique and checking the results of the experiments, it was found that the results did not give much improvement as it was expected. In order to compare the result, we decided to compare the results with an alternative technique in which the CNTs were dispersed in the epoxy at the prior step and then hand laid up on to the reinforcement layers. The average better results were found at 15 to 20 ml of CNT/PAN solution spray, therefore, similar amount of CNTs were selected to be dispersed in epoxy as it was in the grafting technique.

2.4.1. Materials and equipment for preparation of CNT-Epoxy dispersion

Another alternative technique to incorporate the CNTs in the composite system is through epoxy dispersion. The materials and equipment required for preparing the CNT-Epoxy dispersion are listed in this section. The ultrasonic mixer was used in a soundproof casing to avoid ultrasonic waves.

- Epoxy
- Carbon Nanotubes (COOH functionalized)
- Beaker
- Pipette
- Ultrasonic processor with sound proof case
- Weighing Balance

Table 2.3: Material and equipment for CNT-Epoxy dispersion.

Material/Equipment	Specification
CNTs	COOH functionalized MWCNTs from Cheap Tubes Inc. 184 mg/sample
Epoxy	237 g
Ultrasonic Mixer	Heilscher UP 400S (400W)
Sonotrode	H3
Sonication Time	60 min in 2 parts

2.4.2. Procedure for CNT-Epoxy dispersion

In order to compare our results with the grafting technique, the average amount of MWCNTs reinforced in the grafting technique, i.e. for 20ml spray volume, was used to make the CNT-Epoxy dispersion. 237g of epoxy and 184mg of MWCNTs were weighed separately. CNTs were gradually poured in the epoxy and dispersed through Ultrasonication at 50Hz and 40% amplitude. The process was carried out for 30 mins and then it was allowed to cool to avoid temperature build up. After 30 minutes break it was again carried for 30 minutes so that there was no aggregates of CNTs left in the epoxy. This dispersion was then mixed with hardener for hand lay up for making composites.

2.5. Composite Development

2.5.1. Materials and equipment for composite development

The materials and equipment required for making the composite by hand lay-up technique are listed in this section. Composites were prepared on flat glass mould.

- Epoxy Resin
- CNT-Epoxy dispersion
- Hardener
- Glass fibre Reinforcement
- Carbon Fibre Reinforcement
- Glass base (mould)
- Vacuum Pump (figure 2.8)
- Distribution lines
- Vacuum Bagging
- Resin Trap (figure 2.9)
- Flow Mesh
- Peel Ply
- Releaser (lubricant)
- Seal Tape
- Hand Brushes

Figure 2.8: Vacuum pump.

Figure 2.9: Resin trap.

Table 2.4 shows the specifications of the materials that are used for the making of required composites.

Table 2.4: Materials and equipment for composite development.

<table>
<tr><th>Material/Equipment</th><th>Specification</th></tr>
<tr><td>Epoxy Resin</td><td>Resin Layup, 237 gms
Epoxy Resin Specifications
Tensile Strength: 78 - 83 MPa
Tensile Modulus: 3.1 - 3.4 GPa
Elongation at break: 3.5 - 4.0%
Flexural Strength: 113 - 118 MPa
Flexural Modulus: 3.1 – 3.4 Gpa</td></tr>
<tr><td>Hardener</td><td>63 gms</td></tr>
<tr><td>Resin Fibre Ratio</td><td>40:60</td></tr>
<tr><td>Reinforcement</td><td>1- Carbon – Carbon – Carbon (0/90) Unidirectional
2- Glass – Glass – Glass (0/90) Unidirectional
3- Carbon – Glass – Carbon (0/90) Unidirectional
4- Glass – Carbon – Glass (0/90) Unidirectional
Mechanical Properties of Reinforcements
Fabric Type: Carbon; Weave: Plain; Weight (g/m^2): 200; Warp Density (ends/cm): 5; Weft Density (picks/cm): 5
Fabric Type: E-Glass; Weave: Plain; Weight (g/m^2): 200; Warp Density (ends/cm): 4; Weft Density (picks/cm): 3</td></tr>
<tr><td>Mould</td><td>Flat Glass Type</td></tr>
<tr><td>Vacuum Time</td><td>180 Min</td></tr>
<tr><td>Curing Time</td><td>48 hours (Room Temp)</td></tr>
</table>

2.5.2. Layout of vacuum bagging method

The overview of the vacuum bagging system can be seen from the figure 2.8. There is release film plays an important role in releasing out the composite from the mold. The nylon bagging creates the air trap which is taken out of the system by the vacuum pump.

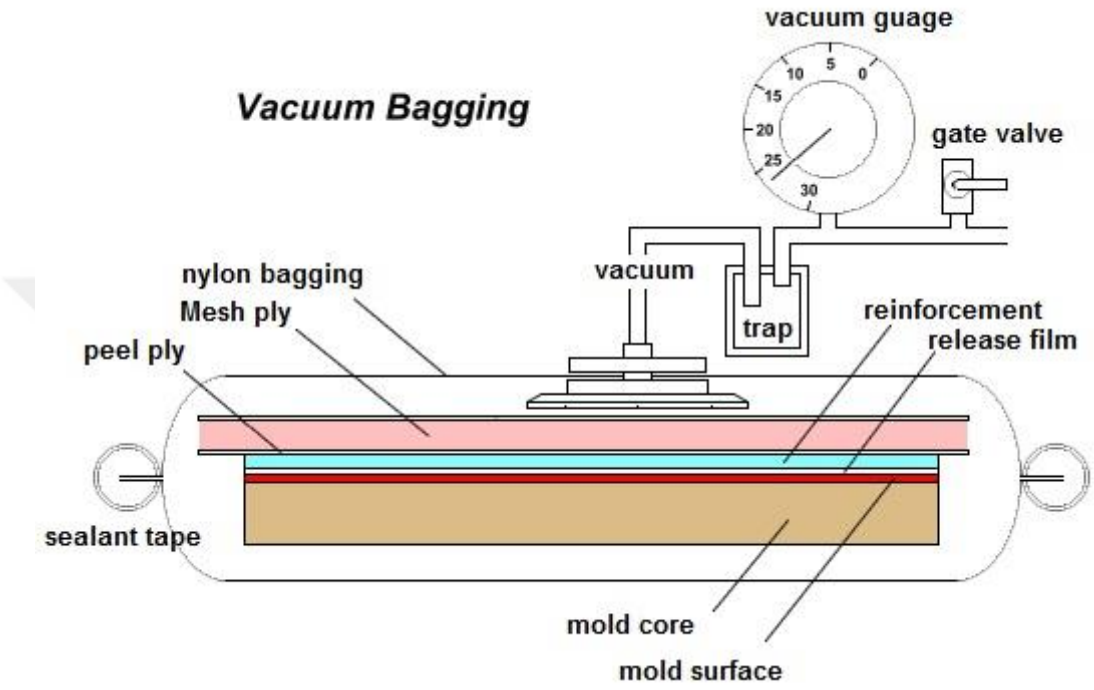

Figure 2.8: Layout for Vacuum Bagging.

2.5.3. Procedure for composite development:

Vacuum Bagging process is a multi-step process starting from mould preparation to finishing of the product. Mould is prepared according to the desired design and shape, polished, cleaned and releaser lubricant is applied. Normally a hand layup skin coat is applied to ensure that there are no voids between the first layer of laminate and the lubricant. Most importantly it reduces the radius of corners which allows the dry glass to be fitted more easily. After drying off, the mixture of resin and hardener is then applied on the lubricant layer by hand layup. Required reinforcements are gradually laid and applied with resin consecutively by homogenous distribution of resin.

The total surface is then covered with peel ply. Peel ply is the only material that separates reinforcement and vacuum. Whole area of the laminate and foam are covered with flow mesh. Flow mesh is restricted from the edges to avoid any possibility of

resin entering the vacuum channel shown in figure 2.10. The resin flow channels are positioned on the top of the laminate. They are wrapped with flow mesh to ensure rapid flow from the channel. The setup is checked patiently to ensure the vacuum integrity.

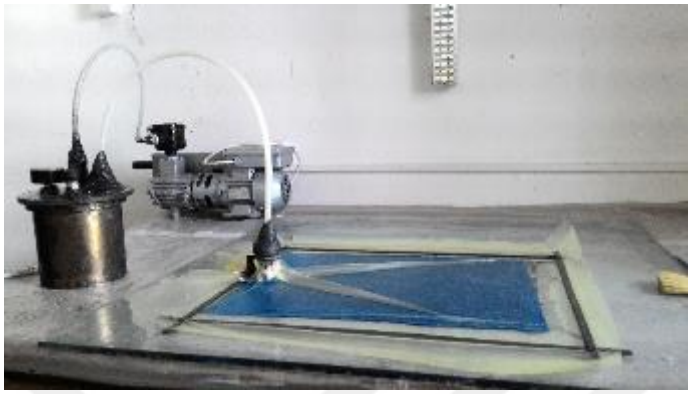

Figure 2.9: Layers of reinforcement covered with mesh flow.

The size of vacuum bag depends on the geometry of the mould. Normally it is taken 1.5 times the length and width of the dimensions of the mould. It should be handled carefully to avoid any damage, as even a small hole can create a lot of trouble. It is recommended to remove all the blades or sharp edged objects from the proximity of the mould to ensure no damage occurs to the bag. The vacuum bag is attached to the mould with the help of sealant tape.

Pleats are introduced to ensure that there is enough film to cover the complex parts of the design and any possible bridging can be avoided by providing excess film. When the vacuum bag is attached completely, vacuum lines are connected. Air is removed from the bag by applying vacuum. If any poor fit is found the vacuum is released and is applied again after some corrections. Figure 2.10 shows the complete system after the vacuum checking.

Figure 2.10: Vacuum check.

A catch pot is used in between the mould and the vacuum pump to avoid any possible entry of resin into the pump. The vacuum level used should be greater than 0.8 bars and as near to 1.0 bar as practical. The bag itself needs to be checked for leaks with leak detectors and all leaks need to be repaired. When no further leaks are found a vacuum drop test should be carried out to ensure that setup is completely sealed. A vacuum gauge is fitted to the bag while the vacuum pump is clamped off.

The setup is given the time for 3 hours to squeeze out the excessive resin after which the vacuum pipe is sealed. After completing the required curing time under room temperature the composite is ready for taking off.

2.6. Tensile Testing

2.6.1. Materials and equipment for tensile testing

After making out the samples, the testing procedure was followed by the cutting of the specimen upto the required standards. Instruments which were used in the tensile tests are mentioned in this section.

- Digital Vernier Calliper for measurement.
- Cutting machine for cutting samples and tabs (figure 2.11).

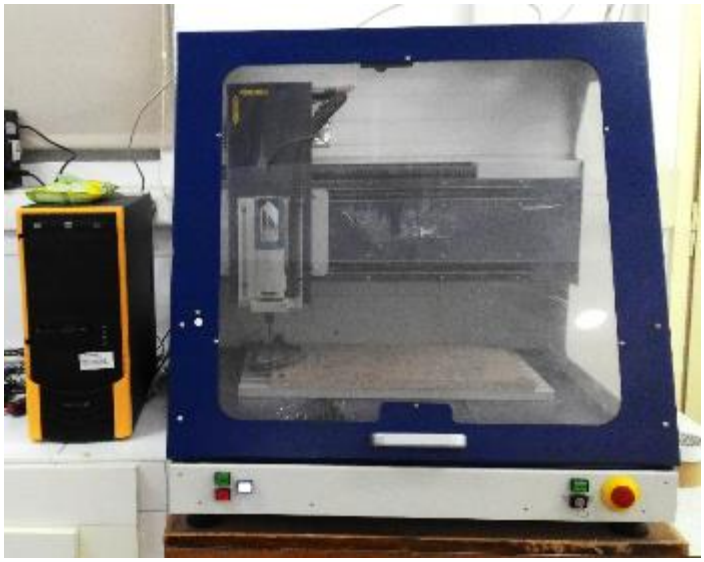

Figure 2.11: CNC machine.

- Tensile Testing Machine by SHIMADZU (figure 2.12) with special assembly was mounted for tensile testing that was designed to grip the sample and pull in longitudinal direction.

Figure 2.12: UTM by SHIMADZU.

- Extensometer to measure the change in length (figure 2.13).

Figure 2.13: Extensometer camera.

- Trapezium Software was used to obtain the raw data and it MS Excel for Data Analysis.

2.6.2. Specimen preparation for tensile test

The test specimen were obtained from laminates. The dimensions of the samples are according to the ASTM standard D-3039. Tabs were attached to the samples, as shown in the figure 2.14, for the successful introduction of load and to avoid the premature failure. The chemical used to bond the tabs to sample is Epoxy.

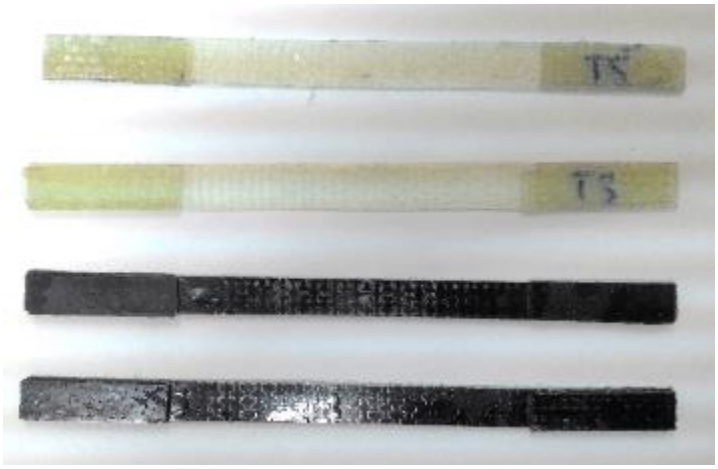

Figure 2.14: Test specimen with tabs.

Table 2.5: Table for specimen dimensions for Tensile Strength.

Sample Type	Thickness, d [mm]	Width, h [mm]	Length of specimen, [mm]	Cross Sectional Area , (A) [mm^2]
				A=d*h
CCC	1.030	14.120	250	14.543
10 CCC	1.040	14.200	250	14.768
15 CCC	0.956	14.192	250	13.568
20 CCC	0.978	14.234	250	13.922
25 CCC	1.060	14.280	250	15.142
CGC	0.920	14.140	250	13.008
10 CGC	1.100	14.254	250	15.680
15 CGC	1.058	14.254	250	15.081
20 CGC	1.098	14.308	250	15.711
25 CGC	1.100	14.633	250	16.117
GCG	0.960	14.680	250	14.082
10 GCG	1.074	14.422	250	15.487
15 GCG	1.080	14.248	250	15.388
20 GCG	1.086	14.228	250	15.451
25 GCG	1.120	14.256	250	15.967
GGG	0.946	16.640	250	15.738
10 GGG	1.194	14.250	250	17.014
15 GGG	1.140	14.122	250	16.097
20 GGG	1.178	14.120	250	16.634
25 GGG	1.156	14.138	250	16.344

2.6.3. Tensile test procedure

- Tensile Tester was switched on and sample was placed in grippers as figure 2.16 shows.
- Alignment was checked and corrected.
- Let the grippers to grab the tabs. The tabs helps in distributing the load and avoid pre mature failure.
- Extensometer was attached to measure the extension or change in length. Position of extensometer is shown in figure 2.15.
- Thickness and width values were measured by Vernier Callipers.

- Thickness and width value are noted for each sample and added to software before the test loading starts.
- The gauge length was observed by putting marking stickers on the specimen and noted by using extensometer.
- The test was carried out at a loading rate of 6 mm/min.
- The unit of measurement of stress is MPA while strain values are dimensionless.
- Mechanical behaviour plot was checked on screen during the tests for any undesirable events like slippage etc.
- As the applied load increases, the sample elongates, in the beginning according to an elastic behaviour and later on cracks start appearing.
- Cracks initiations are visible and the cracking sound can also be heard.
- When the internal stresses reach the maximum resistance values the material fails and breaks which can be seen in figure 2.17.
- The CNT reinforced composites are not ductile materials hence the failure was sudden and exploding break.

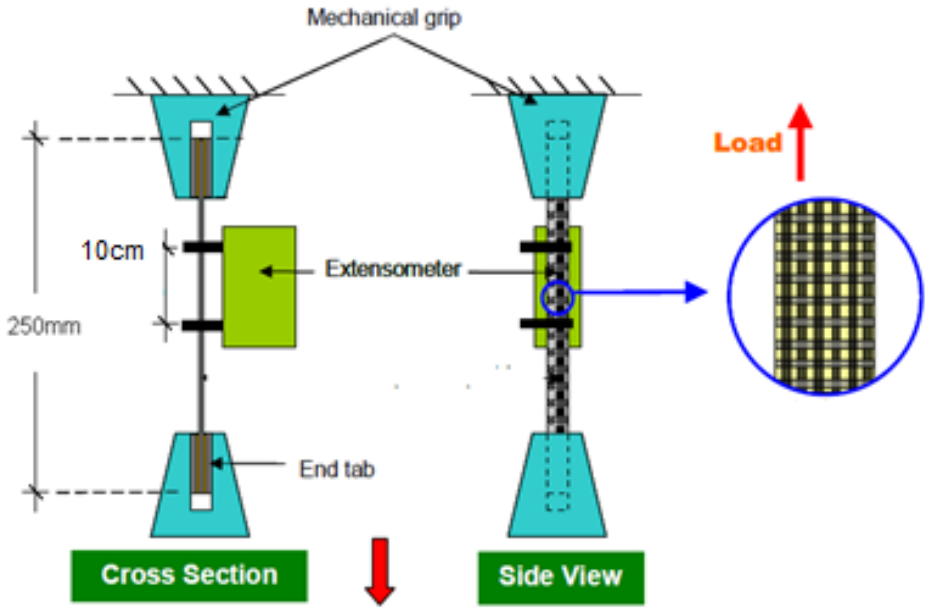

Figure 2.15: Pictorial representation of tensile test.

Figure 2.16: Specimen clamped on tensile tester.

Figure 2.17: Specimen break at maximum load.

The axial tensile stress (σ) is computed at any load level using Equation (2.1) as:

$$\sigma = \frac{P}{A} \quad \textbf{(2.1)}$$

P is the applied load and (A is the cross-sectional area. The corresponding axial strain

ε is computed as a function of the specimen extension (ΔL) and the gauge length (L) using Equation (2.2) as:

$$\varepsilon = \frac{\Delta L}{L} \quad \textbf{(1.2)}$$

The modulus of elasticity E was computed as per ASTM D3039 standard by fitting the tensile chord modulus within 1000 and 3000 micro-strain range as in Equation (2.3):

$$E = \frac{\Delta \sigma}{\Delta \varepsilon} \quad \textbf{(2.3)}$$

Whereas: $\Delta\sigma$ = change in stress, $\Delta\varepsilon$ = change in strain.

2.7. Bending Test

Three point bending test is carried out to determine the flexural Properties of Composites. ISO 14125 standard procedures is followed while performing the experiment. Specimen are subjected to flexure in such a way that the applied load produces curvature of the facing planes of specimen. Test is performed on the mechanical test machine at a crosshead speed of 5 mm/min.

Five specimens from each sample were tested. Load versus displacement values are recorded. Flexural stress (σ) values were determined by using equation (2.4).

$$\sigma_f = \frac{3FL}{2bh^2} \quad \textbf{(2.4)}$$

σ_f is the flexural stress, F is the load, L is the span length, h is the thickness of specimen and b is the width of the specimen.

The flexural modulus is calculated from the equation (2.5).

$$E_f = \frac{L^3}{4bh^3}\left(\frac{\Delta F}{\Delta s}\right) \quad \textbf{(3.5)}$$

E_f is the flexural modulus of elasticity, Δs is the difference in deflection, ΔF is the difference in load.

2.7.1. Material and equipment for bending test

3 point bending test was used to determine the bending strength of the composite material. Equipment used for the bending test are listed below.

- Vernier Callipers
- Mechanical Testing Machine
- Midspan Loading Assembly

2.7.2. Specimen preparation for bending test

Samples are obtained from composites developed by Vacuum bagging technique in rectangular shape (figure 2.18). Dimensions of the samples are according to the ISO 14125.

Figure 2.18: 3-point bending test specimen.

5 specimen were taken from each sample so that the test measurement can be taken from different parts of the sample. It helped in defining the appropriate standard deviation when all of the results were combined. The mean values along with the standard deviation allowed us to understand the working of the system quite easily.

The dimensions of samples (table 2.6) are in accordance with the ISO standard for flexural properties of composites: ISO 14125.

Table 2.6: Table of specimen dimensions for 3 point bending test.

Sample Type	Thickness (mm)	Width (mm)	Span length of Support (mm)	Cross Sectional Area , (A) [mm^2]
CCC	0.896	14.952	40	13.389
10 CCC	1.088	15.234	40	16.519
15 CCC	0.996	14.64	40	14.585
20 CCC	0.94	14.406	40	13.542
25 CCC	1.09	14.696	40	16.015
GGG	0.9	15	40	13.500
10 GGG	1.216	14.488	40	17.613
15 GGG	1.102	14.164	40	15.612
20 GGG	1.156	14.248	40	16.467
25 GGG	1.056	14.506	40	15.320
GCG	0.952	14.538	40	13.846
10 GCG	1.068	14.646	40	15.639
15 GCG	1.094	14.346	40	15.698
20 GCG	1.1	14.256	40	15.686
25 GCG	1.146	14.662	40	16.804
CGC	0.912	14.56	40	13.273
10 CGC	1.08	14.548	40	15.710
15 CGC	1.068	14.318	40	15.293
20 CGC	1.176	14.358	40	16.884
25 CGC	1.1	14.868	40	16.352

2.7.3. Bending test procedure

- Loading fixtures are arranged appropriately following the instructions given by ISO 14125 with the basic principle as in figure 2.19.
- Load is applied to the specimen through steel bar (figure 2.20).
- Figure shows the point bending test arrangement.

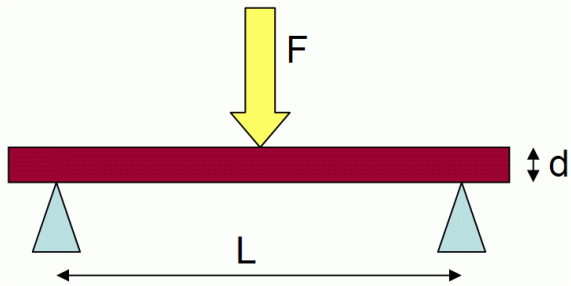

Figure 2.19: Three point bending test.

- Span length is kept according to the standard and it depends on sample length.
- Maximum load and deflection is recorded for the test. The breaking event can be seen in figure 2.21.
- Load vs. displacement curves are plotted to determine stiffness elastic modulus.

Figure 2.20: Midspan loading arrangement.

Figure 2.21: Loading produced deflection in composite.

2.8. Impact testing (Drop weight test)

This test methodology is used to determine the damage resistance of composites facing a drop-weight impact event (ASTM International). ASTM D7136 test standard was used to carry this test. This test is carried by a fall of a mass with a semispherical tip from a certain height onto the laminate. The weight of the impactor was 35 kg. This test was not carried for the standardized specifications since ASTM D7136 specifies a value of 5.50 kg for the impactor, but allows the use of other values. The energy with which the impactor strikes the test specimen is dependent on the mass of impactor and the height from which it is dropped (Justo, et al., 2015). The equation (2.6) defines the calculation of potential energy.

$$E_p = mgh \tag{2.6}$$

Where E_P is the potential energy, m is the mass of the impactor, g is the gravitational acceleration and h is the height from where the impactor is dropped.

Preliminary results from the Charpy test showed that these specimen required around 5 joules of energy to observe impact. In this test the energy was fixed to 5.15 J for our specimens which was attained at a height of 1.5 cm from the surface test specimen. The fixed energy can help in comparing the damage caused by the impact.

2.8.1. Specimen preparation for impact testing

Samples are obtained from composites developed by Vacuum bagging technique in rectangular shape (figure 2.22). Dimensions of the samples are according to the ASTM D7136 standard i.e. 75 mm by 125 mm. The specimen are marked with their centre point.

The most important thing for carrying the drop weight test is that the impact should strike the centre of the specimen. To allow this, the specimen were marked with their centre points with a marker and they were subjected to the concentrated drop weight impacts for the measurements.

Figure 2.22: Specimen for impact testing.

2.8.2. Materials and equipment for impact testing

Drop weight test was used to measure the impact response of the composite material. Equipment and materials used for the impact testing are listed below.

- Impact testing machine
- Impactor (35 Kg)
- Vernier Calliper for measurements
- Rubber grip for holding specimen

2.8.3. Impact testing procedure

- Loading fixtures are arranged appropriately following the instructions given by ASTM D7136 with the basic principle as in figure 2.23.

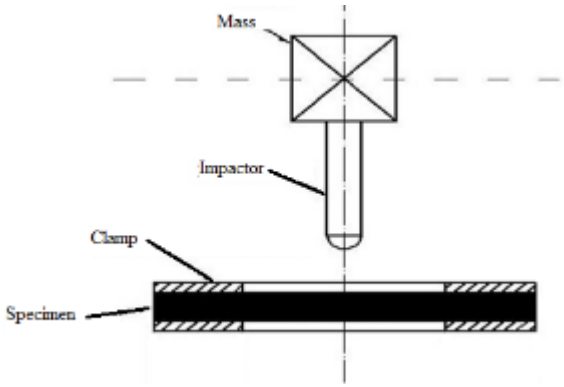

Figure 2.23: Drop weight test mechanism.

- The drop weight equipment used for testing can be seen in figure 2.24. It was being used in the conveyor belt industry for measuring the impact resistance

of reinforced rubber, therefore, some alterations were required to make it useable for our test specimen.

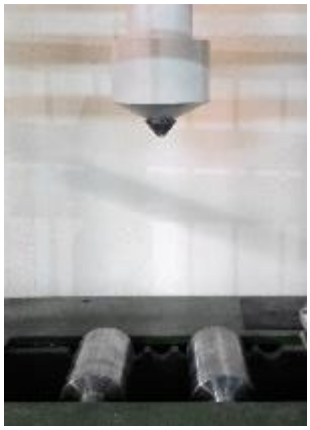

Figure 2.24: Drop weight testing equipment.

- Specimens were marked with their center points as in figure 2.25. It is necessary for the test that the impactor should fall exactly at the center point for adequate results.

Figure 2.25: Impactor falling at the center of the specimen.

- After the impact there is some damage and dent caused around the center of the specimen which helps in measuring and comparing the impact response.

3. RESULTS AND CONCLUSION

3.1. Tensile testing

3.1.1. Ultimate tensile strength of CNT grafted composites

After taking the strength measurements from the testing equipment the mean values of the 5 tests for each sample were plotted in Table 3.1.

Table 3.1: Ultimate tensile strength of CNT grafted composites.

Sample Type	Extensometer Gauge Length, Lg, [mm]	Cross Sectional Area , (A) [mm^2]	Maximum Load Before Failure, (P_{max}) [N]	Extensometer Displacement, (δ), [mm]	Ultimate tensile Strength, (F_{tu}) [MPa]	Standard Deviation
		A=w*h	$P_{max} = F_{tu}*A$		$F_{tu}=P_{max}/A$	
CCC	100.582	14.543	7205.002	1.117	495.691	27.255
10 CCC	100.664	14.768	7454.688	1.040	507.602	58.752
15 CCC	100.286	13.568	7539.376	0.880	555.696	22.843
20 CCC	101.652	13.922	6632.500	0.883	477.266	28.108
25 CCC	100.696	15.142	6752.814	0.977	447.689	38.679
CGC	100.460	13.008	5516.876	1.136	424.870	33.547
10 CGC	100.020	15.680	5881.500	1.150	375.516	26.362
15 CGC	100.262	15.081	5838.438	1.402	387.294	54.023
20 CGC	100.976	15.711	6261.812	1.261	399.340	25.798
25 CGC	100.580	16.117	6256.770	1.055	392.936	78.380
GCG	100.613	14.082	4317.735	1.135	306.907	23.083
10 GCG	100.190	15.487	4387.254	1.154	283.393	15.669
15 GCG	100.364	15.388	4474.066	1.211	291.363	37.551
20 GCG	101.124	15.451	4256.566	1.203	275.498	10.275
25 GCG	99.866	15.967	4597.878	1.267	288.196	6.726
GGG	100.394	15.738	4075.626	1.662	259.908	43.330
10 GGG	100.870	17.014	3602.126	1.562	211.853	9.264
15 GGG	101.228	16.097	3129.690	1.503	194.556	10.374
20 GGG	100.936	16.634	3352.816	1.567	201.956	19.306
25 GGG	100.966	16.344	3842.438	1.571	234.839	27.952

3.1.2. Tensile strength plots for CNT grafted composites

Stress-strain curves for the CNT grafted specimen as compared with the non-grafted specimen are plotted in below graphs (figure 3.1 to 3.4).

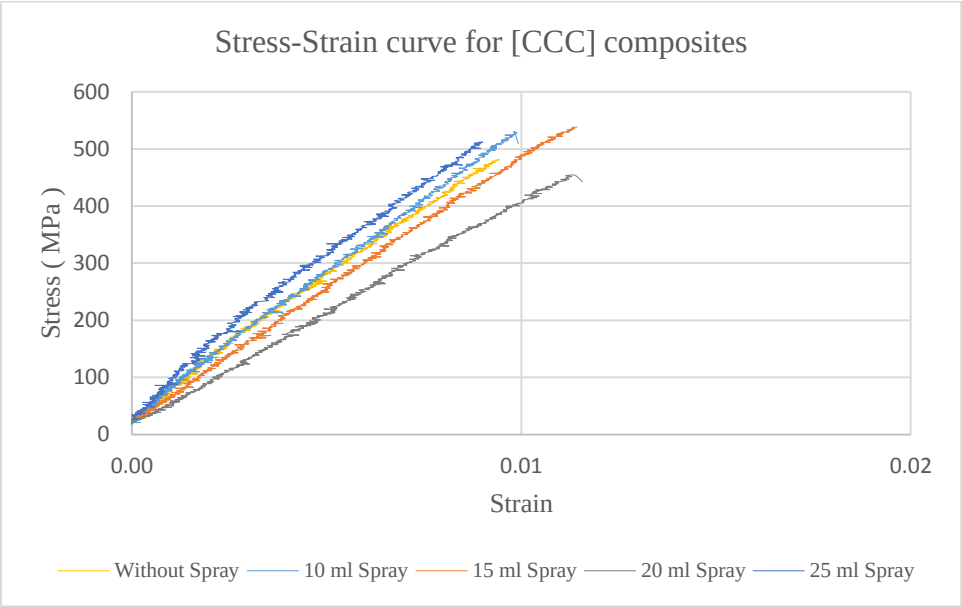

Figure 3.1: Stress-strain curve for [CCC] composites (CNT grafted).

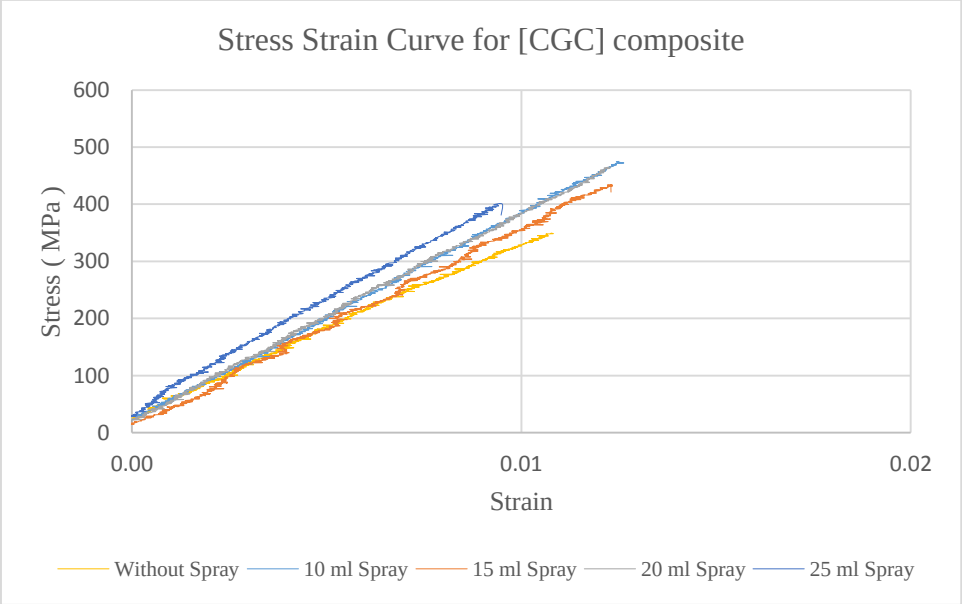

Figure 3.2: Stress-strain curve for [CGC] composites (CNT grafted).

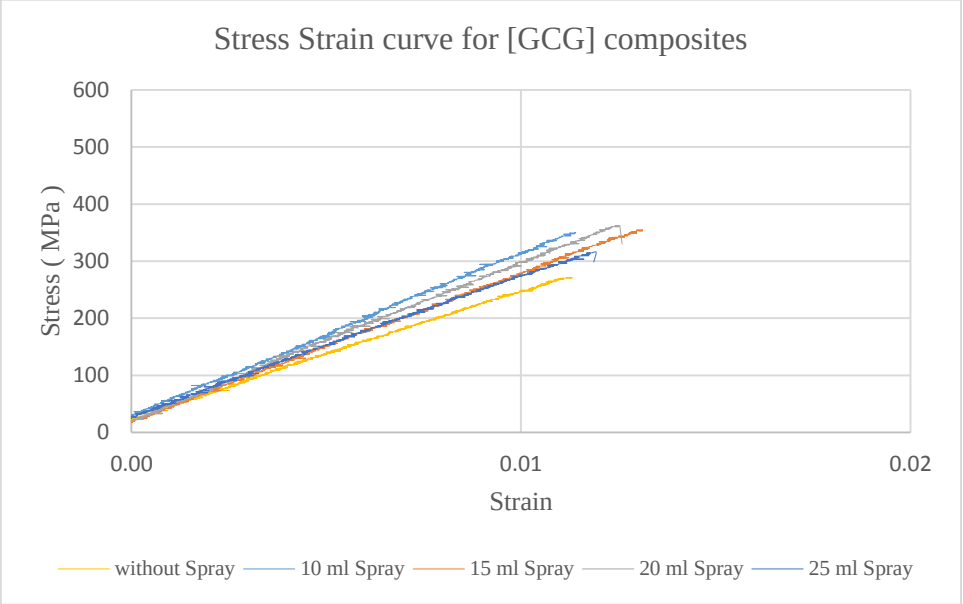

Figure 3.3: Stress-strain curve for [GCG] composites (CNT grafted).

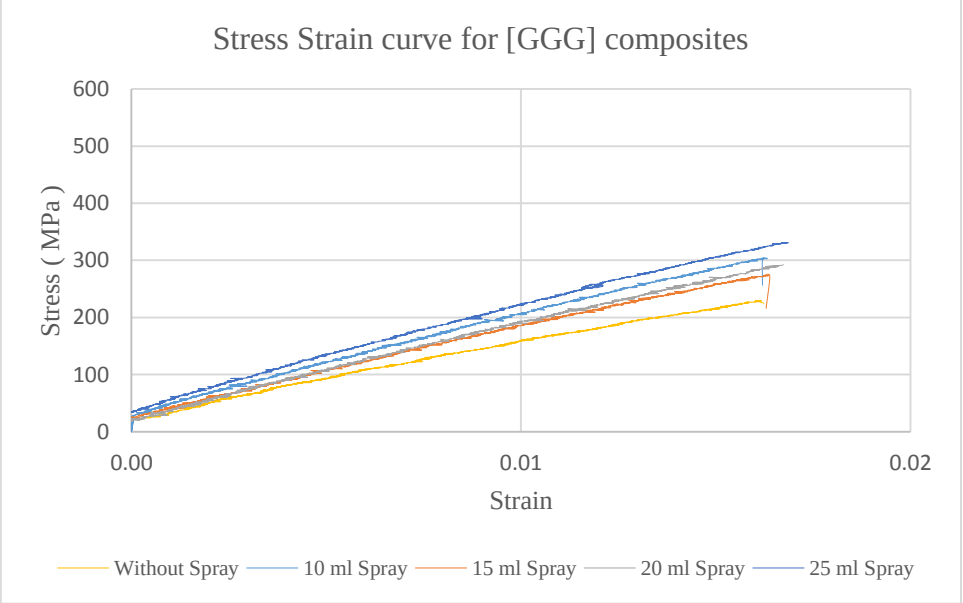

Figure 3.4: Stress-strain curve for [GGG] composites (CNT grafted).

3.1.3. Tensile strength comparison plots for CNT grafted composites

The mean tensile strength of the CNT grafted specimen are compared with the non-grafted specimen are plotted in the graphs below (figure 3.5 to figure 3.8).

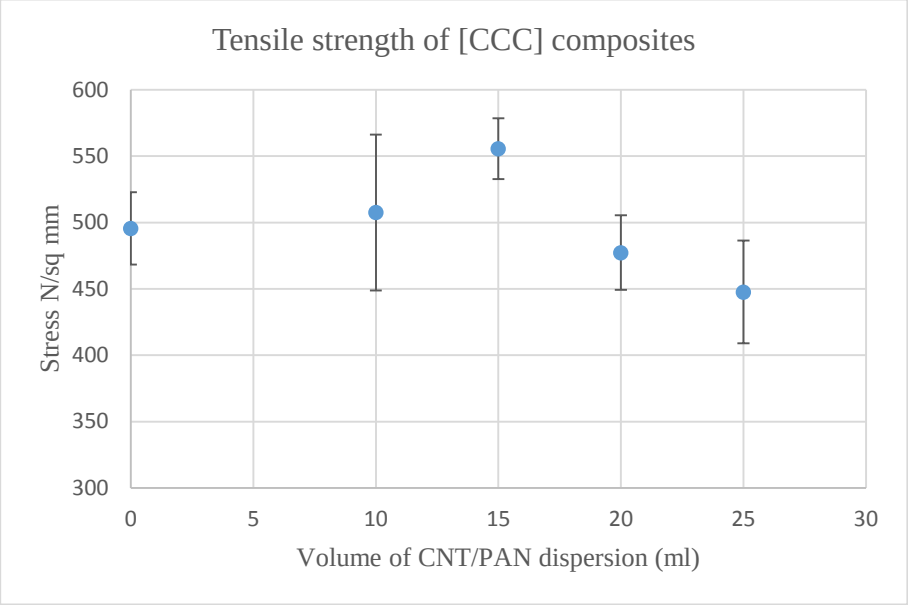

Figure 3.5: [CCC] composites comparison plot for the tensile strength (CNT grafted).

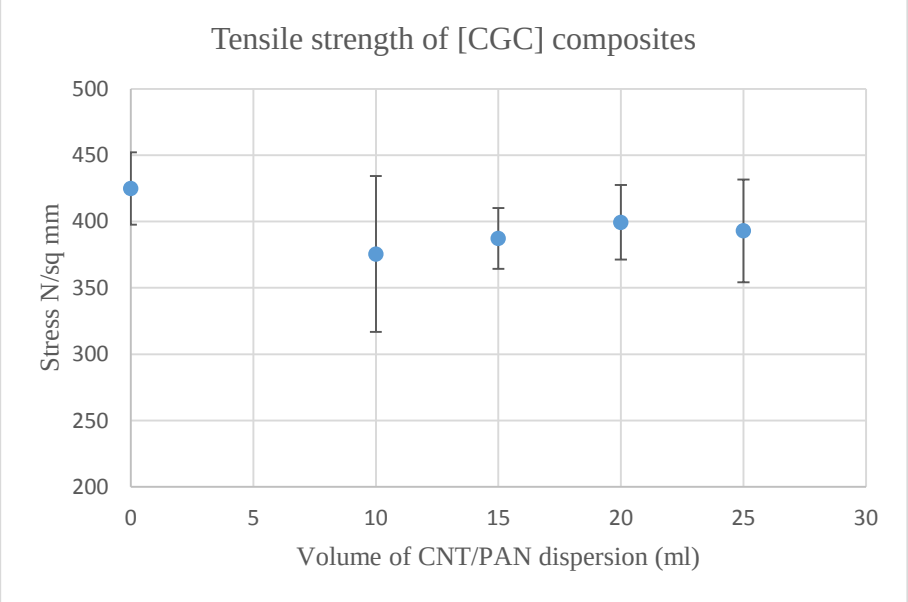

Figure 3.6: [CGC] composites comparison plot for the tensile strength (CNT grafted).

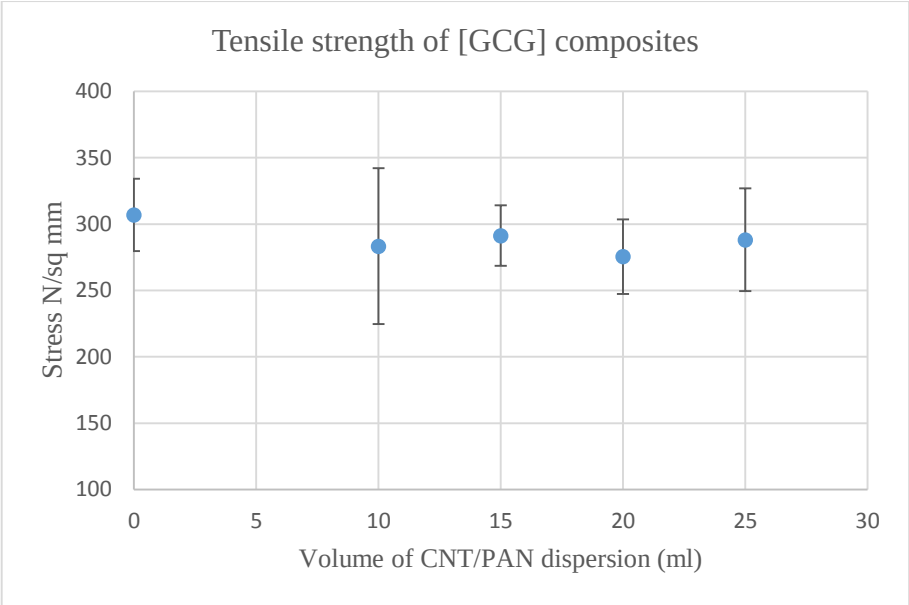

Figure 3.7: [GCG] composites comparison plot for the tensile strength (CNT grafted).

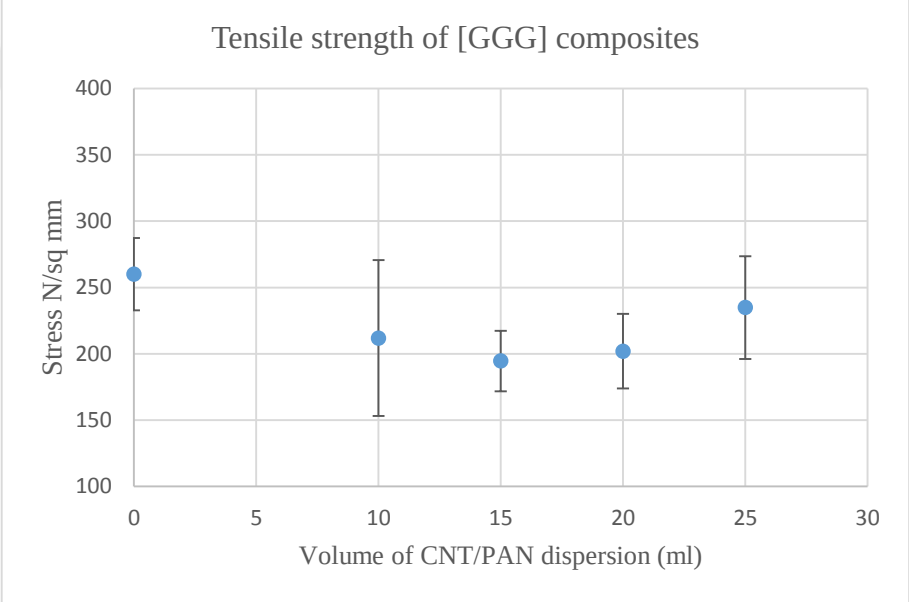

Figure 3.8: [GGG] composites comparison plot for the tensile strength (CNT grafted).

3.1.4. Tensile results analysis for CNT grafted composites

- The results from table 3.1 showed some variations in the results around the mean values of tensile strength which is due to the non-homogenous distribution of the matrix and CNTs.
- The ultimate tensile strength of three layers of carbon grafted with 15 ml of CNTs on each layer showed the maximum results followed by the 10 ml of CNT/PAN solution on carbon-carbon-carbon composite (figure 3.5). There was a significant increase in the tensile strength while increasing the CNT/PAN grafting on reinforcements from 0 ml to 15 ml. Some of the CNTs would have successfully grafted onto the carbon fibres to create network structure resulting in increased tensile property (Lee, et al., 2015).
- From the figure 3.5 to figure 3.8 it is observed that the MWCNTs grafted composites didn't show much significant improvement in the ultimate tensile strength of the composites as expected. The result was constant except for the two categories. This may be due to the unsuccessful bonding between the MWCNTs and carbon/glass fibres (Lee, et al., 2015).
- Three layered non-grafted Glass fabric composite achieved the maximum elongation before breaking whereas the three layered carbon composite with 25 ml of CNT/PAN solution grafted on them showed the lowest elongation before breaking (figure 3.4). The MWCNTs present also work as fillers which impart stiffness to the final product. Glass fibre is already less stiffer than the carbon fibre due to which this result can be judged easily (Christine, n.d.).
- Results showed that the higher gauge lengths in [CCC] composites showed the least elongation before breaking whereas in the [GGG] composites this behaviour was opposite. The difference in the stiffness property of glass and carbon fibre attributes to this effect (Christine, n.d.).
- The tensile test results shows that the load increased to the maximum value and then dropped suddenly confirming a brittle fracture for the studied composites (figure 3.1 to figure 3.4). Reason for this behaviour is the Matrix-reinforcement interaction which makes the resultant material brittle through chemical bonding (Lubineau & Rahaman, 2012).
- It has been observed that the crack propagates in a direction perpendicular to the direction of the external load, delamination with explosion was also

observed in [GCG] composites. Since carbon fibre have more stiffness as compared to the glass fabric, it produced some sliding action causing the delamination of the outer layers of glass fabric causing a bursting effect. It can be said that the low tensile strain bearing ability of carbon fibre directs the load concentration on the middle layer resulting in prior breakage (Wisnom, et al., 2015).

- From the stress-strain curves we can observe that the composites that are grafted with higher quantities of MWCNTs show higher modulus. Higher quantities of MWCNTs make the composite stiffer resulting in a more brittle behaviour. Grafted CNTs on the reinforcements create a networking structure which increase the rigidity of the material and helps in avoiding the shear cracking (Ma & Kim, 2011).

3.1.5. Ultimate tensile strength of CNT-Epoxy dispersed composites

The mean tensile strength results of the composite samples prepared by the dispersion technique are plotted in the table 3.2.

Table 3.2: Ultimate tensile strength of CNT-Epoxy dispersed composites.

Sample Type	Extensometer Gauge Length, Lg, [mm]	Cross Sectional Area , (A) [mm^2]	Maximum Load Before Failure, (P_{max}) [N]	Extensometer Displacement, (δ), [mm]	Ultimate tensile Strength, (F_{tu}) [MPa]	Standard Deviation
CCC	100.530	13.808	7330.210	2.077	531.127	51.999
CGC	99.460	13.744	6383.333	2.442	465.260	30.956
GCG	99.207	13.676	4660.417	2.481	341.009	20.071
GGG	98.623	13.634	3203.127	2.867	234.974	5.176

3.1.6. Stress Strain curves for CNT-Epoxy dispersed composites

Stress-strain curves for the CNT dispersed specimen as compared with the non-CNT specimen are plotted in below graphs (figure 3.9 to 3.12).

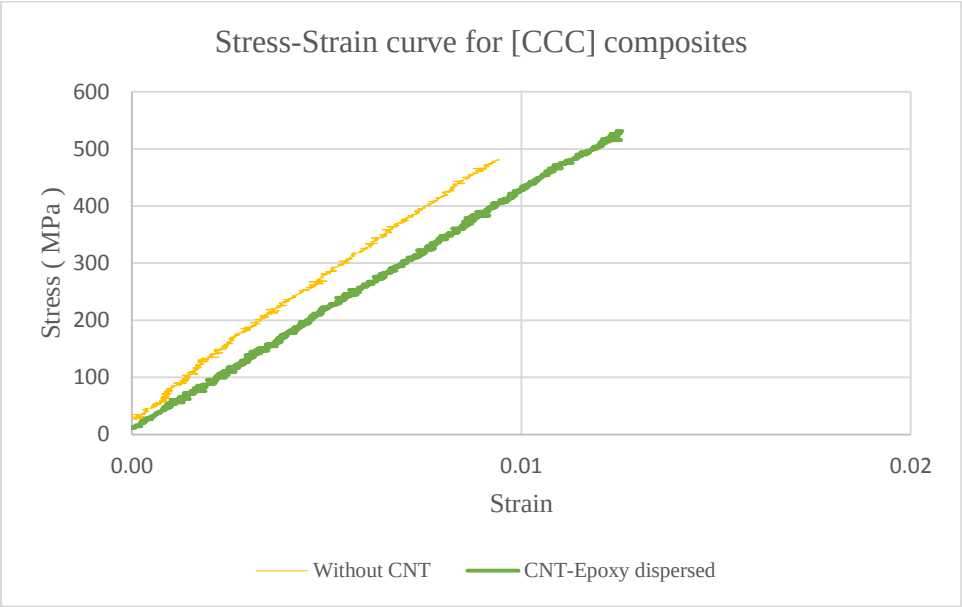

Figure 3.9: Stress-strain curve for [CCC] composites (CNT-Epoxy Dispersed).

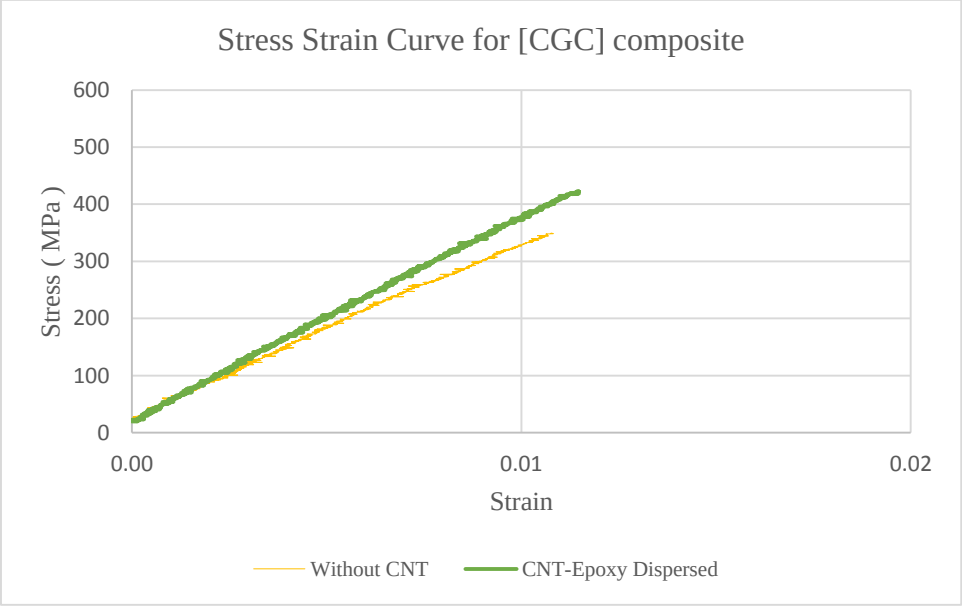

Figure 3.10: Stress-strain curve for [CGC] composites (CNT-Epoxy Dispersed).

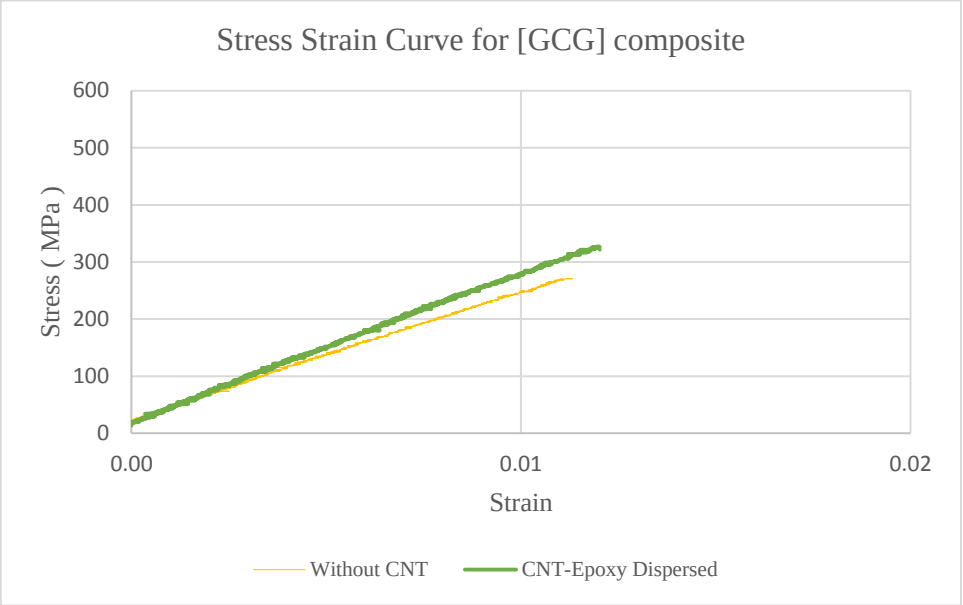

Figure 3.11: Stress-strain curve for [GCG] composites (CNT-Epoxy Dispersed).

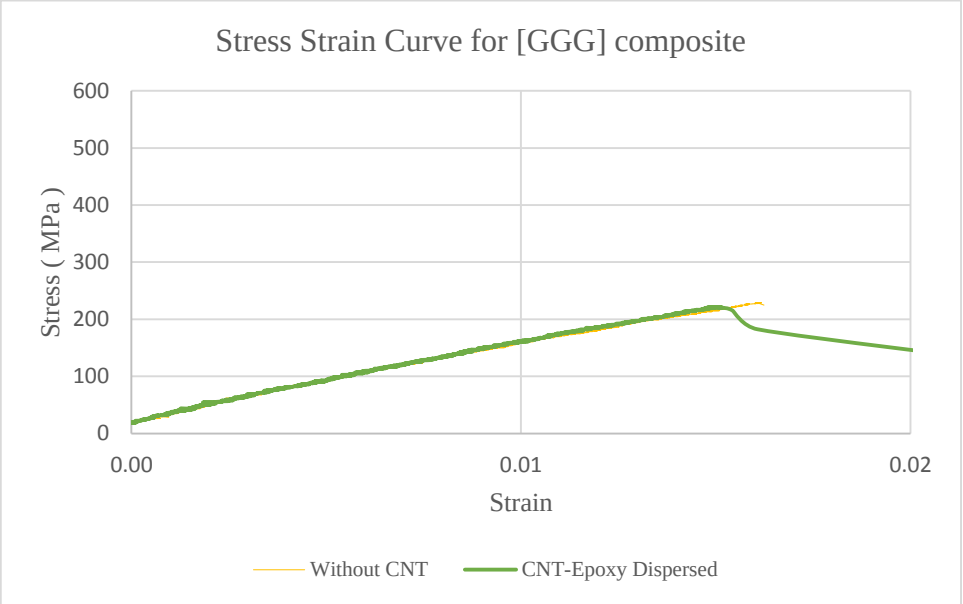

Figure 3.12: Stress-strain curve for [GGG] composites (CNT-Epoxy Dispersed).

3.1.7. Tensile results analysis for CNT-Epoxy dispersed composites

- Tensile strength of the CNT-Epoxy dispersed composites was measured by UTM. Table 3.2 shows the test results which were compared with the composites prepared without CNT.
- From figure 3.9 to figure 3.12, the stress strain curves show the variations in the maximum stress capacity, strain and modulus values of the composites.
- It can be observed from the curves that there was an increase in the maximum stress and strain values with a slightly increased modulus except for the [GGG] composites prepared with CNT-Epoxy dispersion.
- Observing that the desired tensile properties were not achieved with the grafting technique we prepared 4 specimen with the same combinations using MWCNT-Epoxy dispersion with the same amount of MWCNTs that we used for 20ml CNT/PAN dispersion. Results were compared with the highest results for MWCNT grafted composites in each of the combinations as shown in figure 3.13.

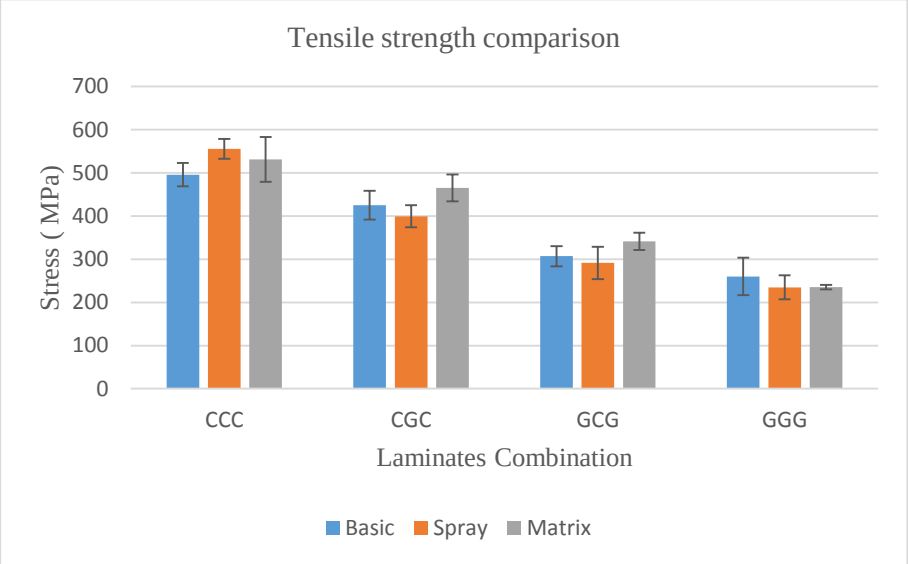

Figure 3.13: Tensile strength comparison of different technique.

- When we compare the highest tensile strength results from each category of the composites in accordance with the Basic composites, Spray coated/grafted composites and the composites in which matrix was reinforced with MWCNTs, we can see that there is no significant increase in the tensile

strength except for the [CCC] composite in which the strength was increased up to 12%. In other categories the tensile strength was approximately same.

- Figures 3.14 to 3.17 show the comparison of stress- strain curves for within different techniques. It can be observed from the curves that there is a slight improvement for CNT grafted [CCC] composites but other categories were almost same.

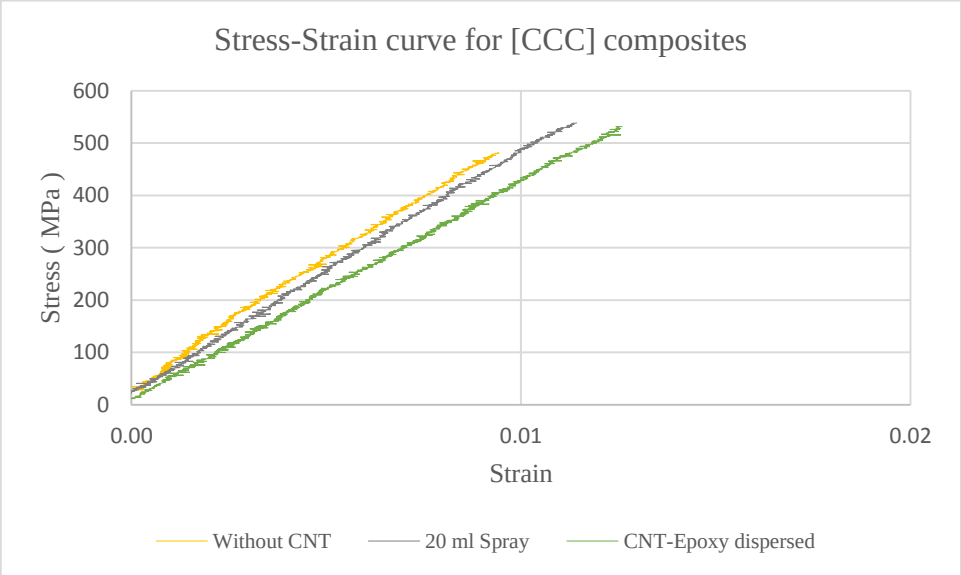

Figure 3.14: Comparison of stress-strain curve in different techniques for [CCC] composites.

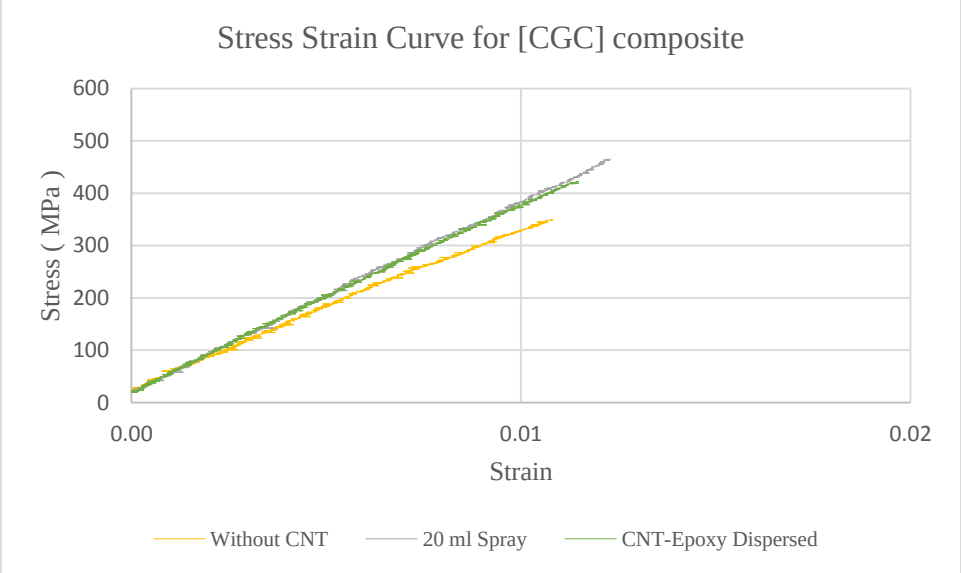

Figure 3.15: Comparison of stress-strain curve in different techniques for [CGC] composites.

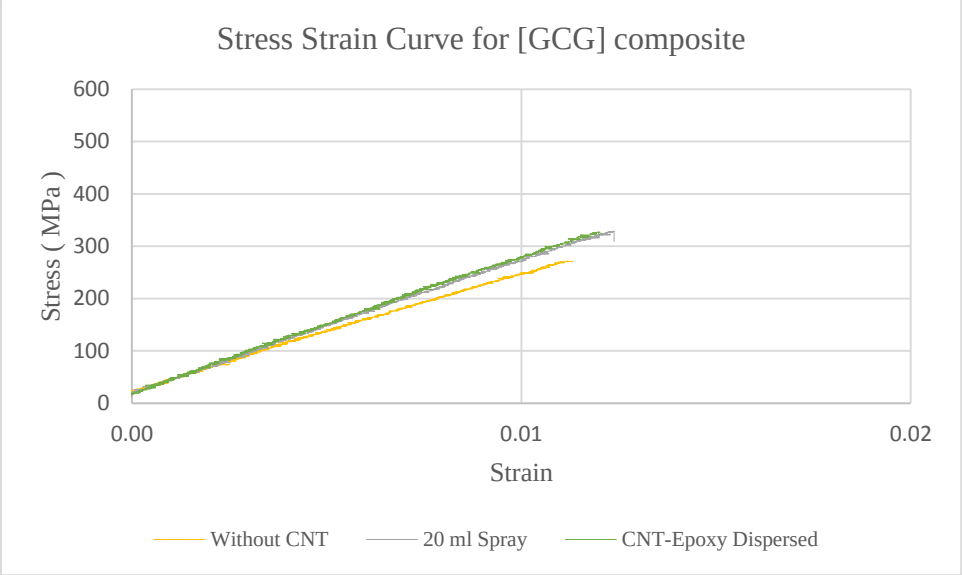

Figure 3.16: Comparison of stress-strain curve in different techniques for [GCG] composites.

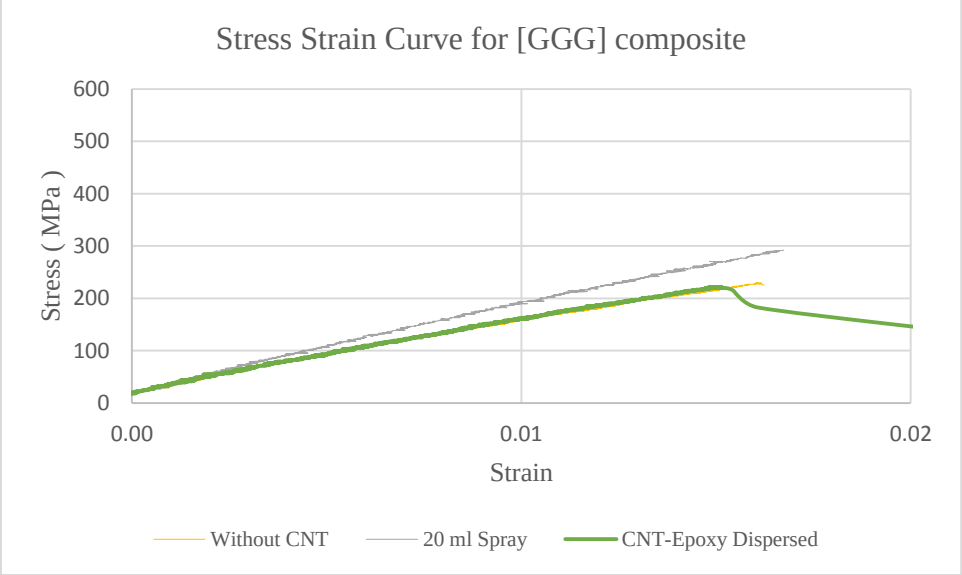

Figure 3.17: Comparison of stress-strain curve in different techniques for [GGG] composites.

- Composites influenced by the CNTs have anisotropic properties, in which the alignment of the CNTs play an important role. The mechanical properties along the dimension of the CNTs are enhanced whereas the properties dependent on the perpendicular direction are sacrificed (Ma & Kim, 2011).
- The size of the CNTs helps in stacking the CNTs in composite material. Smaller diameter CNTs encourage more aligned stacking in the matrix, therefore, imparting more mechanical properties (Ma & Kim, 2011).

3.2. Three Point Bending Test

3.2.1. Bending test results for the CNT grafted composites

The measurements from the three point bending test were taken for the CNT grafted and non-grafted samples. The mean of 5 values for every specimen is written in table 3.3.

Table 3.3: Bending (flexural) strength of CNT grafted composites.

Sample Type	Span length of Support	Cross Sectional Area , (A) [mm^2]	Max Force (kN)	Flexural Strength N/sq. mm	Standard Deviation	Max Strain	Elastic Modulus (Mpa)
CCC	40	13.389	0.083	414.875	29.089	1.712	26899.920
10 CCC	40	16.519	0.122	409.950	54.123	1.899	13545.358
15 CCC	40	14.585	0.106	435.982	27.912	2.029	24008.600
20 CCC	40	13.542	0.076	356.682	31.247	1.540	25049.780
25 CCC	40	16.015	0.108	371.642	27.079	2.240	17977.700
GGG	40	13.500	0.041	202.994	42.713	2.279	6247.366
10 GGG	40	17.613	0.050	141.388	29.607	2.383	5391.900
15 GGG	40	15.612	0.052	182.178	36.071	2.287	7276.576
20 GGG	40	16.467	0.080	251.010	20.923	2.974	9823.730
25 GGG	40	15.320	0.055	203.578	29.367	2.355	10016.832
GCG	40	13.846	0.040	184.065	38.621	2.618	4072.540
10 GCG	40	15.639	0.062	221.059	29.705	2.989	8707.848
15 GCG	40	15.698	0.065	227.582	29.871	2.795	8973.108
20 GCG	40	15.686	0.058	203.382	19.590	2.557	8707.680
25 GCG	40	16.804	0.060	184.045	16.828	2.624	8279.982
CGC	40	13.273	0.053	263.629	20.567	1.212	23333.920
10 CGC	40	15.710	0.092	324.693	28.154	1.552	22317.060
15 CGC	40	15.293	0.085	311.061	30.263	1.437	23584.840
20 CGC	40	16.884	0.074	230.535	83.845	1.467	17088.856
25 CGC	40	16.352	0.084	280.776	49.722	1.583	20463.380

3.2.2. Bending test plots for CNT grafted composites

Figure 3.18 to figure 3.21 shows the load vs displacement curves of the mean values of the bending test samples of CNT grafted samples compared with the non-grafted samples.

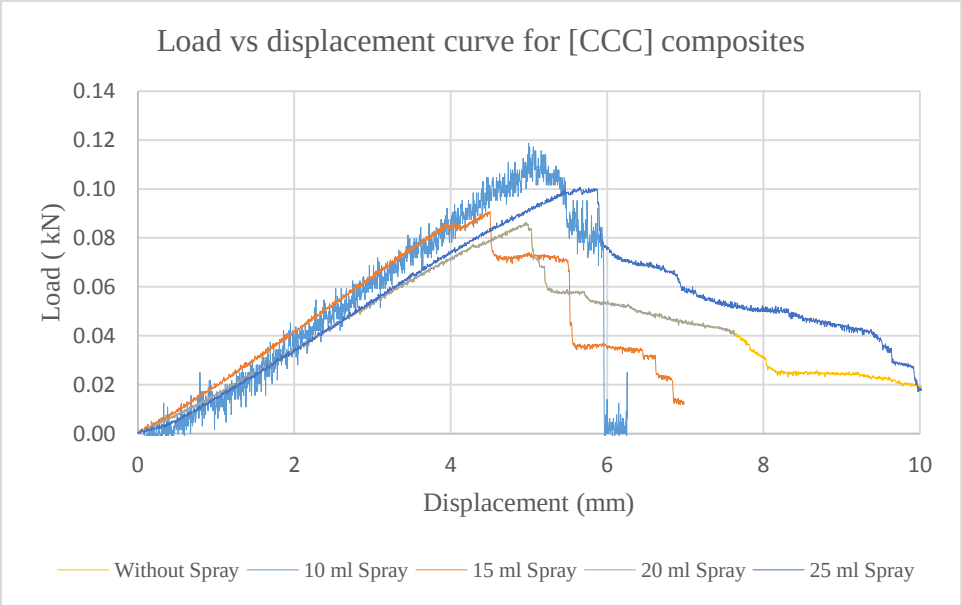

Figure 3.18: Load vs displacment curve for [CCC] composites (CNT grafted).

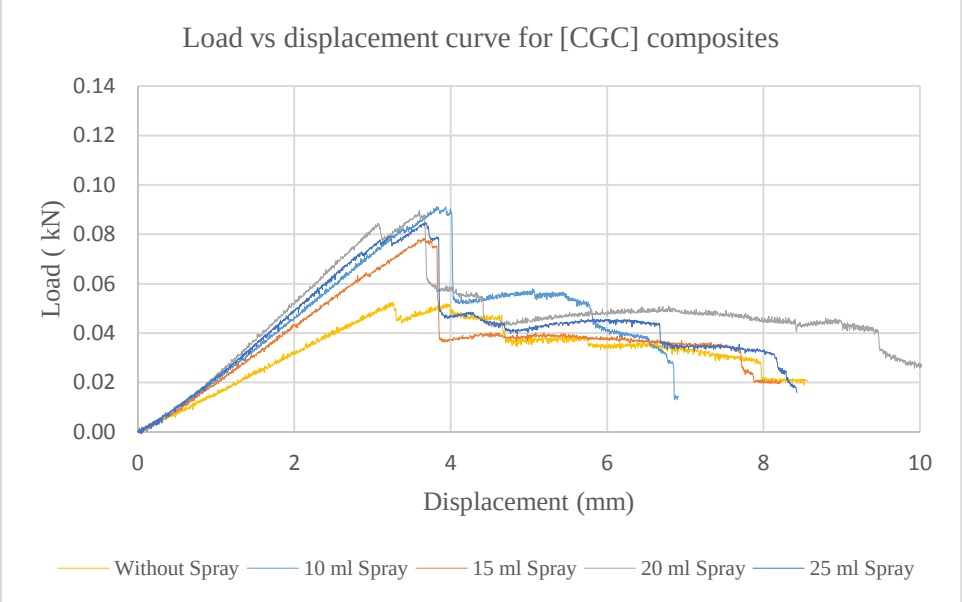

Figure 3.19: Load vs displacment curve for [CGC] composites (CNT grafted).

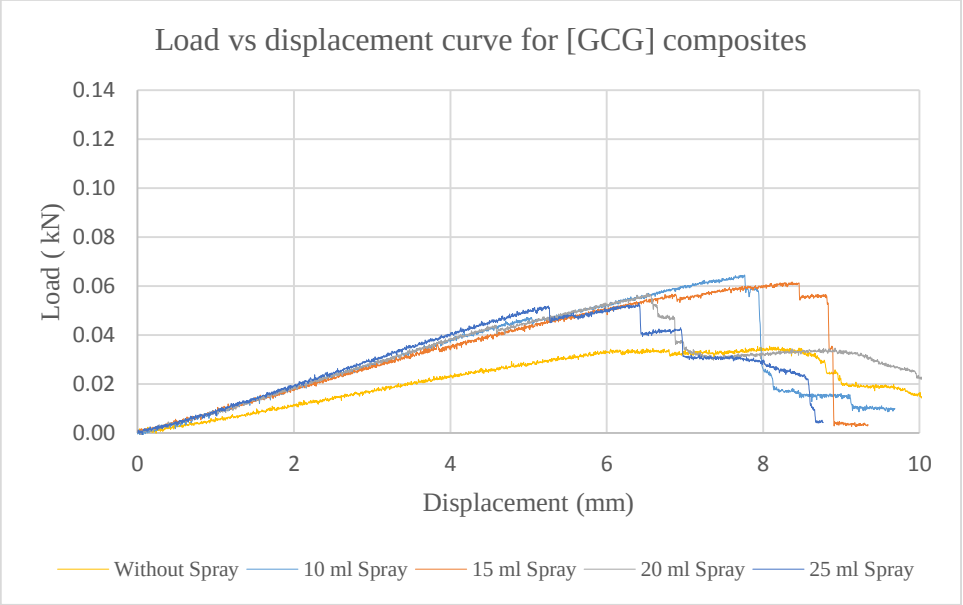

Figure 3.20: Load vs displacment curve for [GCG] composites (CNT grafted).

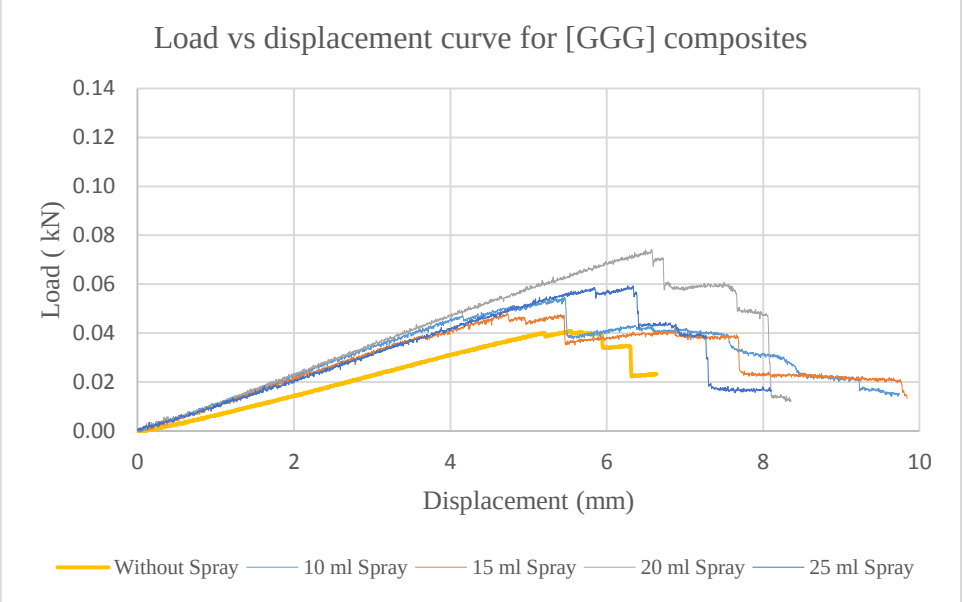

Figure 3.21: Load vs displacement curve for [GGG] composites (CNT grafted).

3.2.3. Bending strength comparison plots for CNT grafted composites

Figure 3.22 to figure 3.25 shows the mean values of the flexural strength of CNT grafted samples compared with the non-grafted sample.

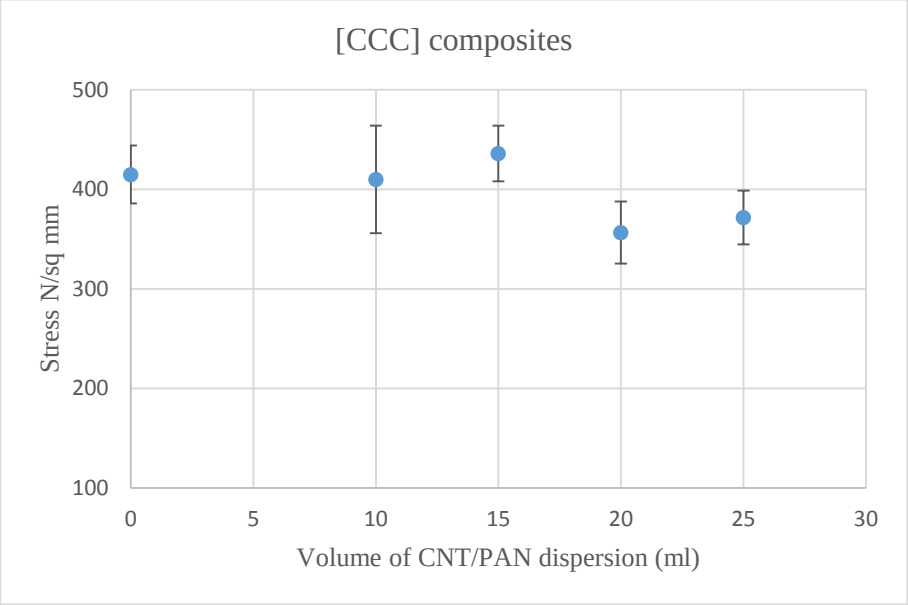

Figure 3.22: [CCC] composites plot for flexural strength (CNT grafted).

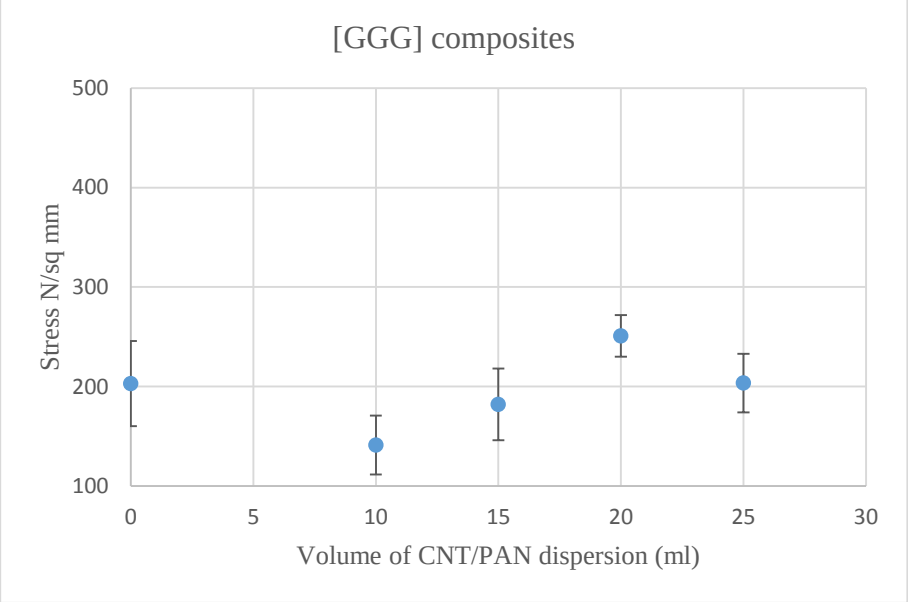

Figure 3.23: [GGG] composites plot for flexural strength (CNT grafted).

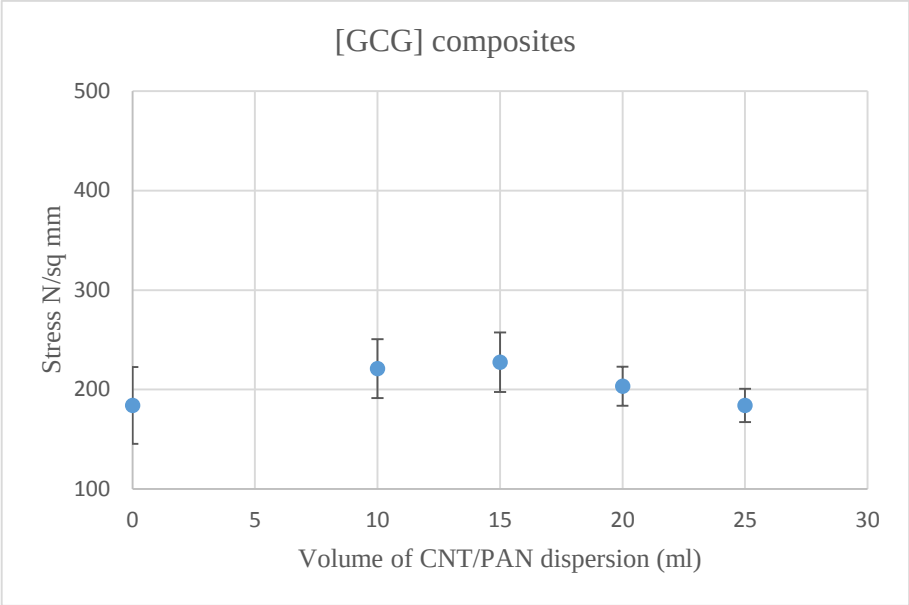

Figure 3.24: [GCG] composites plot for flexural strength (CNT grafted).

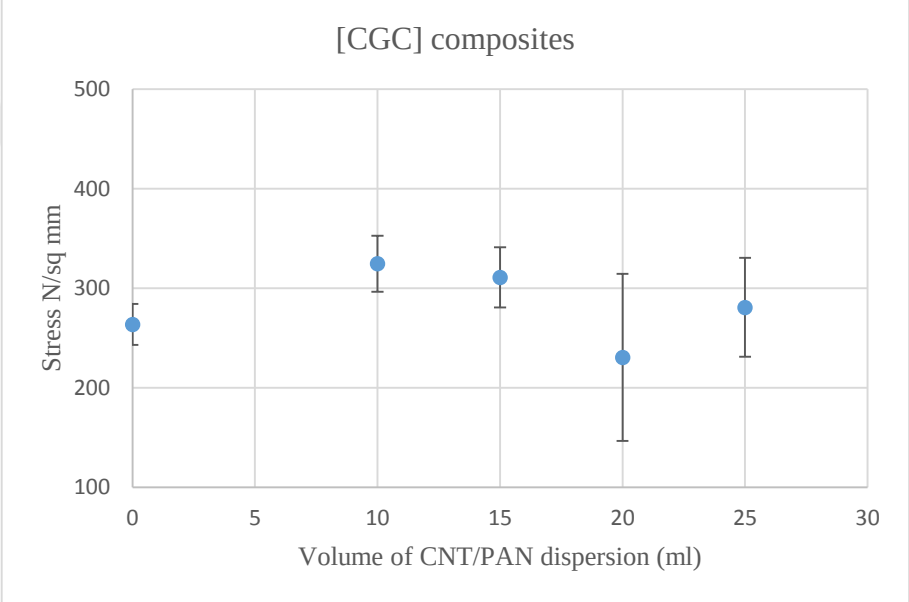

Figure 3.25: [CGC] composites plot for flexural strength (CNT grafted).

3.2.4. Bending test results analysis for CNT grafted composites

- Bending test results from table 3.3 shows that there was a certain improvement in the flexural strength of the CNT grafted specimens.
- In [CCC] composite, 15 ml grafted CNT/PAN showed prior results, [GGG] composites had more flexural strength with 20 ml grafted CNT/PAN, [CGC] composites had better results with 15 ml of CNT/PAN grafted and [CGC] composites showed better results grafted with 10 ml of CNT/PAN solution as shown in figure 3.22 to figure 3.25. The flexural properties are enhanced due to the strong interaction within the matrix structure up to optimum quantity of CNTs. When the CNTs exceed the optimum value they make agglomerates with each other producing certain points where stress can be concentrated to result in premature breakage (Ma & Kim, 2011).
- Results showed an improvement of 23-24% in the flexural strength of the composites except [CCC] composites which showed relatively low improvement.
- The interface of fibre–matrix in the CNTs grafted carbon and glass fibre composite is modified due to change in fibre surface. Further to the interface modification, CNTs produce an interwoven network around the fibre resulting in a nano-composite system which protects the fibre from crack propagation and debonding ensuring a strengthened matrix.
- Flexural strength of the composites decreased with higher quantities of CNT/PAN solution as shown in figure 3.22 to figure 3.25. The reason would be the slippage of the layers of MWCNTs. The primary layers grafted on to the reinforcement had avoided the secondary layers to make strong grafts which had created a slippage behaviour in them. This is due to the reason of agglomeration of consecutive layers of CNTs which produces weak points for concentrated stress (Ma & Kim, 2011).
- The load vs displacement plot of the composite shows that there is a significant increase in the loading capacity of the composites that are grafted with MWCNTs, increasing their flexural strength up to 24% (figure 3.18 to figure 3.21).
- Strain values before breaking also increased significantly with the higher quantities of MWCNTs in the composites. This effect was observed minimum

in the [CCC] composites and it was highest in the [GCG] composites. Since the stiffness of the carbon fibre is higher than that of the glass fibre, therefore, higher glass percentage in the composites have shown higher developments in sharing the flexural improvements.

3.2.5. Bending test results for the CNT-Epoxy dispersed composites

The measurements from the three point bending test were taken for 5 specimen for each sample made by dispersion technique. The mean of 5 values for every specimen is written in table 3.4.

Table 3.4: Bending (flexural) strength of CNT-Epoxy dispersed composites.

Sample Type	Span length of Support	Cross Sectional Area , (A) [mm^2]	Max Force (kN)	Flexural Strength N/sq. mm	Standard Deviation	Max Strain	Elastic Modulus (Mpa)
CCC	40	13.846	0.103	488.476	40.572	1.906	26527.160
CGC	40	13.500	0.087	418.623	28.420	1.673	26379.860
GCG	40	13.273	0.070	321.697	16.994	3.408	11982.580
GGG	40	13.389	0.046	228.881	31.838	2.773	9758.338

3.2.6. Stress Strain curves for CNT-Epoxy dispersed composites

Figure 3.26 to figure 3.29 shows the load vs displacement curves of the mean values of the bending test samples of CNT-Epoxy dispersed samples compared with the non-CNT samples.

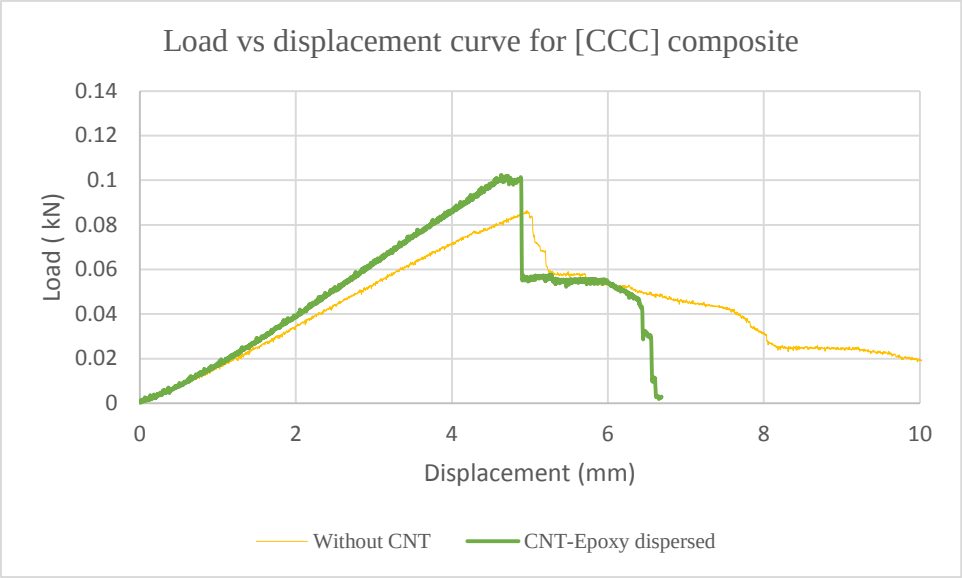

Figure 3.26: Load vs displacement curve for [CCC] composites (CNT-Epoxy dispersed).

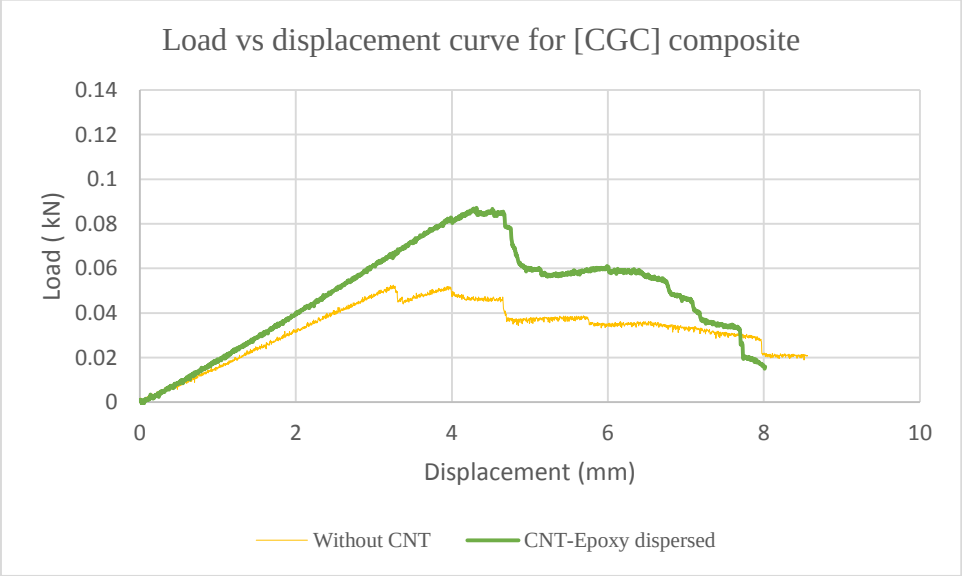

Figure 3.27: Load vs displacement curve for [CGC] composites (CNT-Epoxy dispersed).

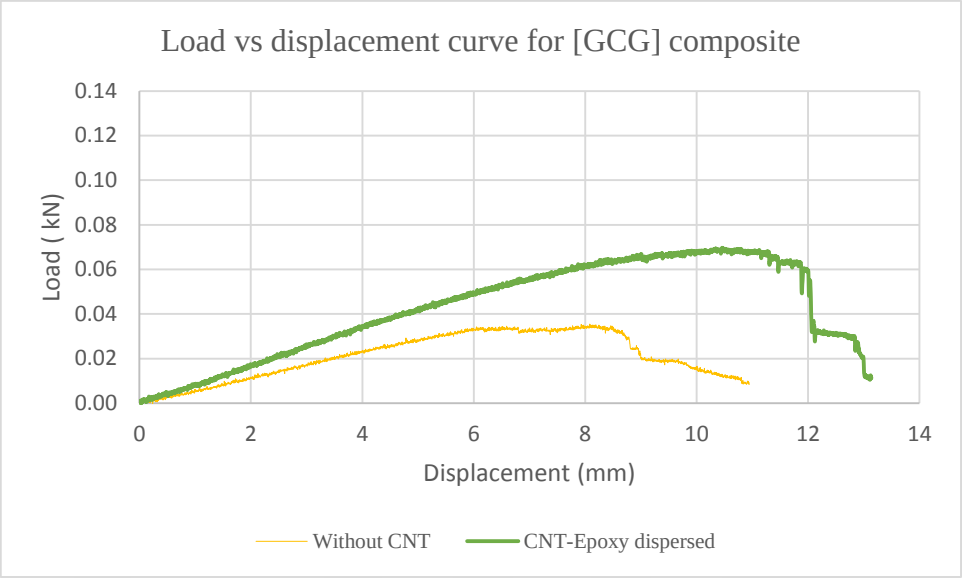

Figure 3.28: Load vs displacement curve for [GCG] composites (CNT-Epoxy dispersed).

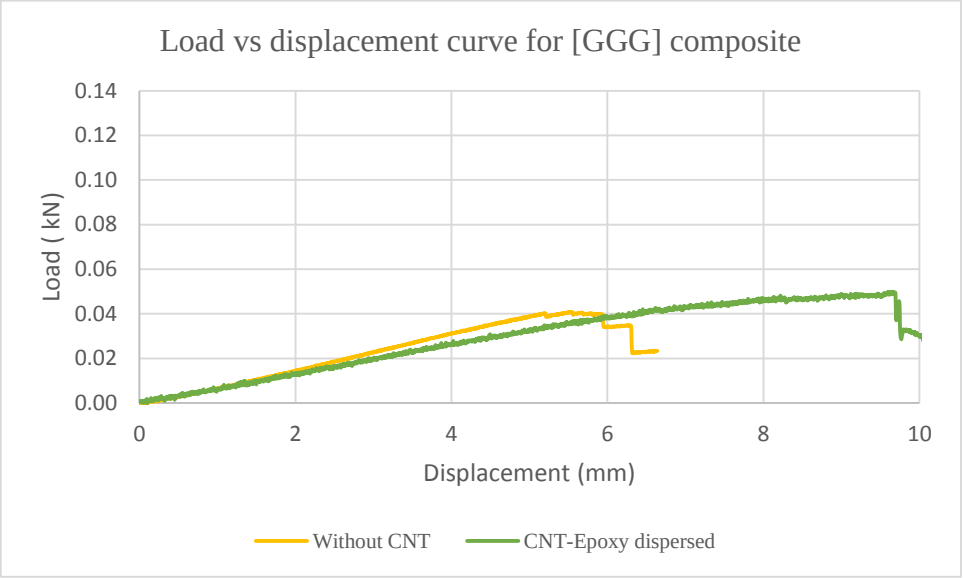

Figure 3.29: Load vs displacement curve for [GGG] composites (CNT-Epoxy dispersed).

3.2.7. Bending test results analysis for CNT-Epoxy dispersed composites

- The flexural strength of the CNT-Epoxy dispersed composites was measured by 3 point bending test. Table 3.4 shows the test results which were compared with the composites prepared without CNT.
- From figure 3.26 to figure 3.29, a significant increase in the loading capacity of the composite can be seen along with the increased strain values. The CNT

networking in the matrix supports the long chains of epoxy, therefore, it spreads the applied load.

- The maximum load was attributed to the [CCC] composites with CNT-Epoxy dispersion as shown in figure 3.26 but the maximum increase was observed in [GCG] composites with CNT-Epoxy dispersion and also the elongation values were the highest for this category.
- On comparison of the composites having the highest flexural strength in each category, we can observe a significant increase in the flexural strength of the grafted composites as well as the composites which were made by dispersing the MWCNTs in the epoxy matrix. The highest improvement is noticed for the [GCG] Matrix dispersed CNTs composite that shows 74% increase in the flexural strength as shown in figure 3.30.

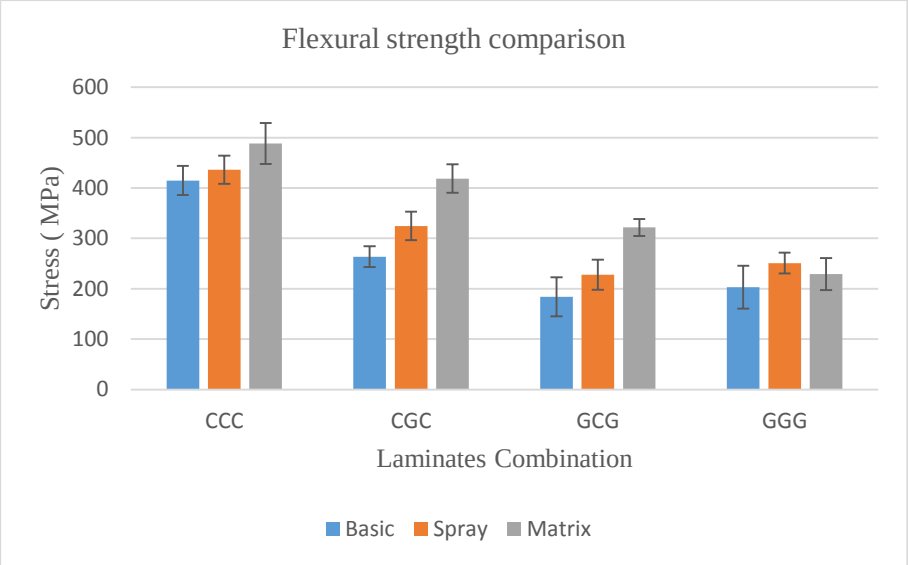

Figure 3.30: Flexural strength comparison of different techniques.

- The sonication of MWCNTs in epoxy caused the proper stacking arrangement and MWCNT-Epoxy interaction. The load transferring ability of the matrix was improved causing an increased flexural strength of the resultant composite (Lubineau & Rahaman, 2012).
- Figures 3.31 to 3.34 show the load vs displacement comparison of the composites manufactured by different techniques. It can be seen that the maximum load was taken by the [CCC] composites influenced with the CNTs. whereas the maximum strain was for the [GCG] and [GGG] composites since glass fibers are less stiff than carbon fibers.

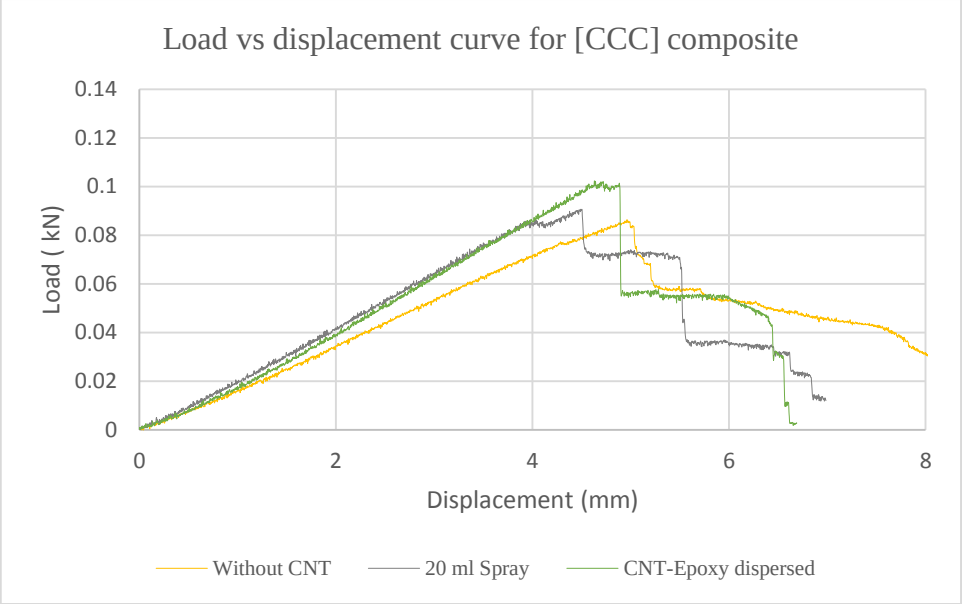

Figure 3.31: Comparison of load vs displacement curves of different techniques for [CCC] composites.

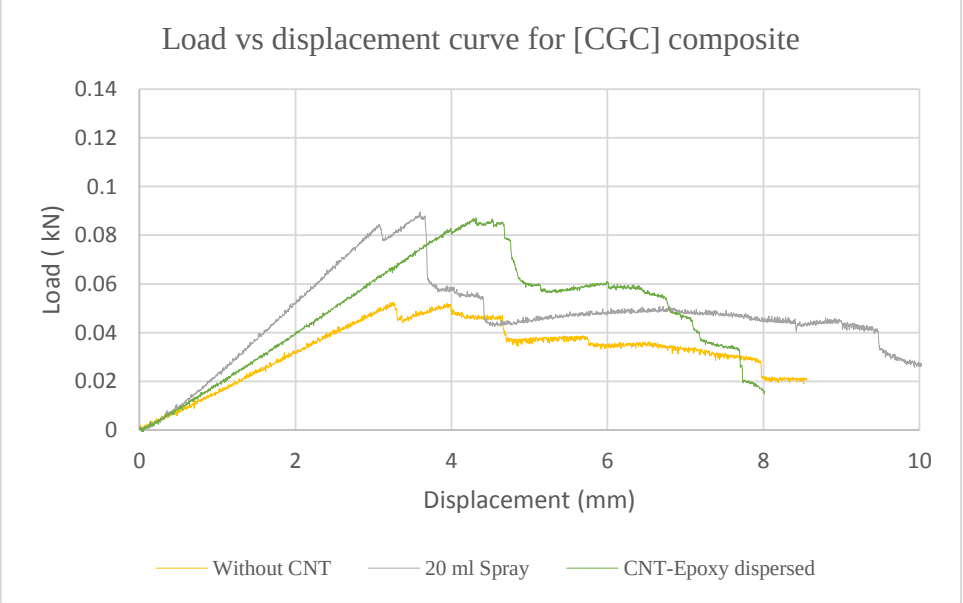

Figure 3.32: Comparison of load vs displacement curves of different techniques for [CGC] composites.

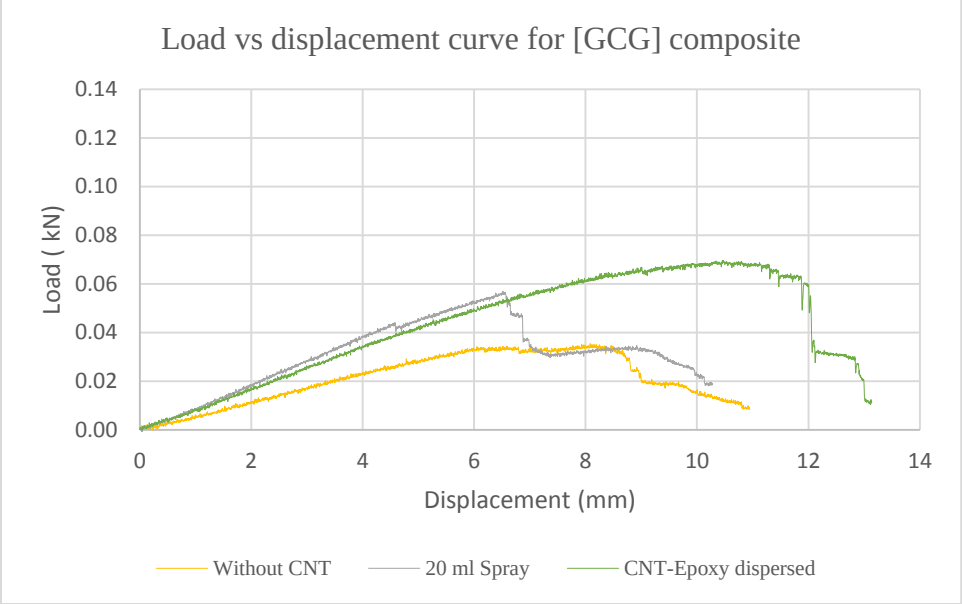

Figure 3.33: Comparison of load vs displacement curves of different techniques for [GCG] composites.

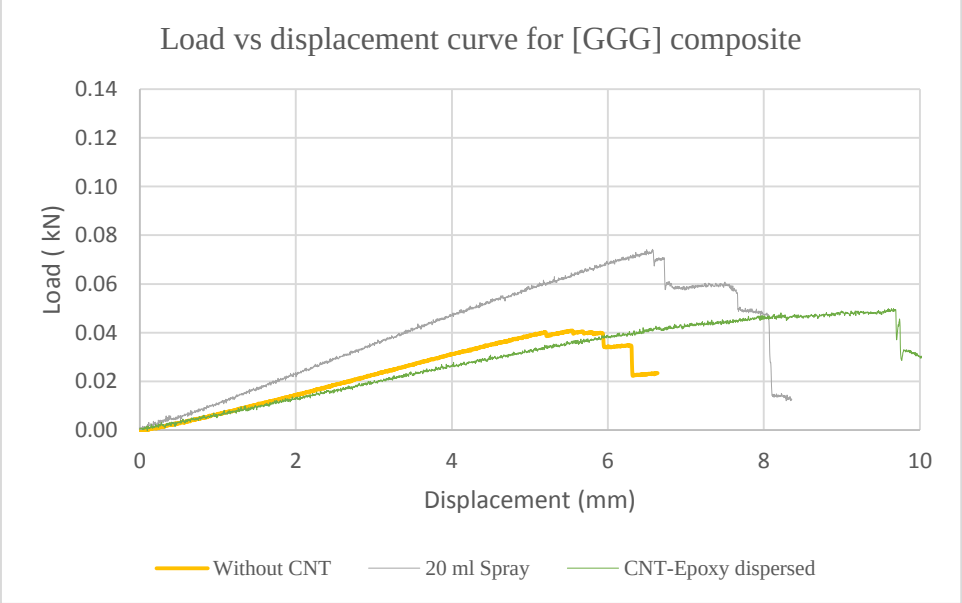

Figure 3.34: Comparison of load vs displacement curves of different techniques for [GGG] composites.

3.3. Impact Strength Test

For the impact response the test were conducted under same amount of energy i.e. 5.15J, as mentioned in the experimental section. The impactor stroked the specimen at their centre from the height of 15mm to gain the specified amount of energy. The damage and the indentation caused by the impactor was analysed as the impact response of the composites.

3.3.1. Impact strength test results for CNT grafted composites

- Figure 3.35 shows the impact response for the [CCC] composites grafted with MWCNTs. From the below picture it can be clearly seen that the initial impact strength of the [CCC] composite without CNTs interaction is not good since the specimen broke in a direction after the impact. The influence of CNTs in the spray volume of 10ml and 15ml show some good properties in distributing the impact shock along axis resulting in less damage. The spray volumes of 20ml and 25ml show an aggressive damage.

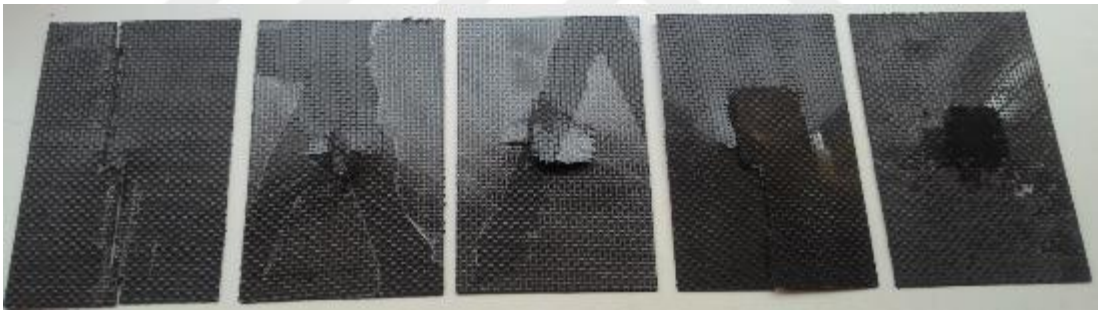

Figure 3.35: Impact response of [CCC] composites (without CNT, 10 ml, 15 ml, 20 ml, 25 ml)

- Figure 3.36 show the impact strength results of the [CGC] composites. As compared with the sample treated without CNTs the spray volumes of 20ml show better results with the less damage.

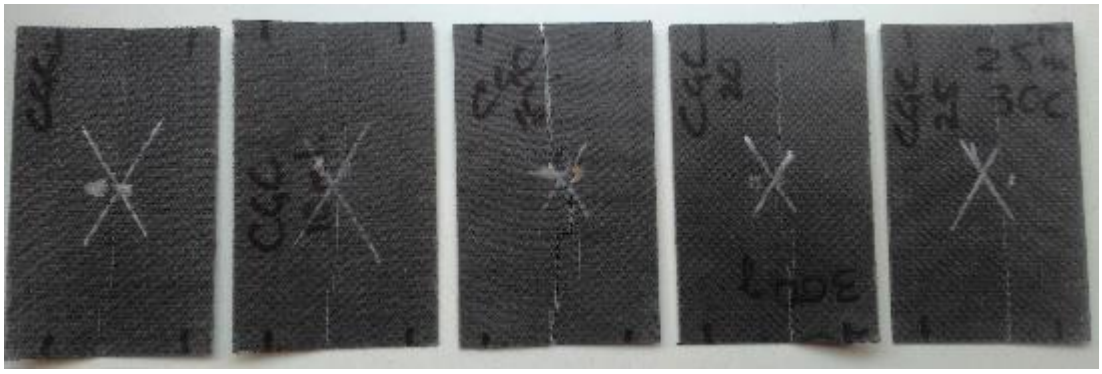

Figure 3.36: Impact response of [CGC] composites (without CNT, 10 ml, 15 ml, 20 ml, 25 ml).

- Figure 3.37 is attributed for the impact results of [GCG] composites. Here also there was a slight improvement in the impact properties of the samples grafted with higher spray volume of CNTs.

Figure 3.37: Impact response of [GCG] composites (without CNT, 10 ml, 15 ml, 20 ml, 25 ml).

- Figure 3.38 shows the impact results of [GGG] composites. The results from the 20ml spray volume of grafted CNTs show good results as compared with the others.

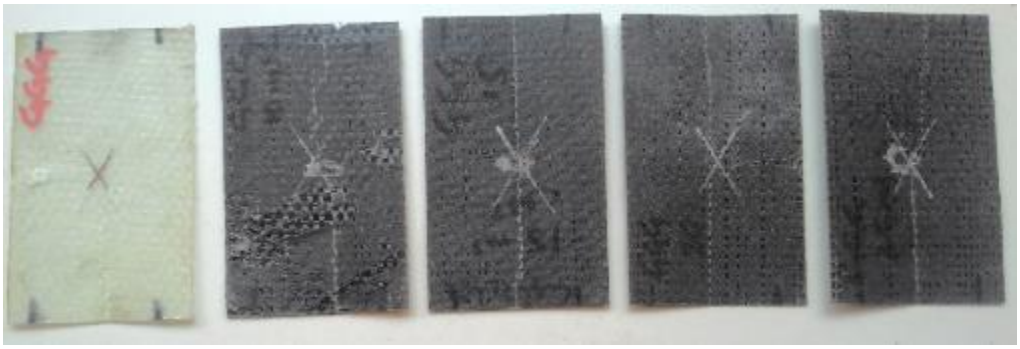

Figure 3.38: Impact response of [GGG] composites (without CNT, 10 ml, 15 ml, 20 ml, 25 ml).

3.3.2. Impact strength analysis for CNT grafted composites

- The impact strength results for [CCC] composites grafted by CNTs showed comparatively less damage area in the 10ml and 15 ml of CNT/PAN grafting. There is more damage that can be seen in the composite without CNT grafting in figure 3.35. Less damaged area can be attributed to the higher bonding ratio of the matrix chains which help in distributing the impact in dimensions along the specimen direction.
- The addition of glass fiber in the [CGC] composites resulted in overall less area of damage. The breakage was less as compared to [CCC] composites. This is because of the better flexural properties of the glass fibers and their less stiff property as compared to carbon fibers (Kalantari, et al., 2015).
- The damage in the composites manufactured by the introducing glass fabric resulted in specific direction i.e. across the length of the specimen. It happened because of the weaving design of the glass fabric in which there were less picks.
- The higher amounts of CNTs tends to make agglomerates on the composite surface which disturbs a proper distribution of the applied impact in all directions. There are points on which the stress becomes concentrated resulting in a damage at premature stage (Ma & Kim, 2011).
- From figures 3.18 to 3.21, it can be observed that the loading capacity of the composites had improved by the interaction on CNTs which also supports the reason for having increased impact properties.

3.3.3. Impact strength test results for CNT-Epoxy dispersed composites

- The samples manufactured by the CNT-Epoxy dispersion were tested in this section under a fixed amount of impact energy i.e. 5.15J. The impactor was dropped from the height of 15mm to gain specified amount of energy.
- Figure 3.39 to 3.42 show the impact result comparison of the different combinations of composites made by CNT-Epoxy dispersion and without CNT. The damage area has reduced significantly as compared with the samples manufactured without CNTs interaction. .

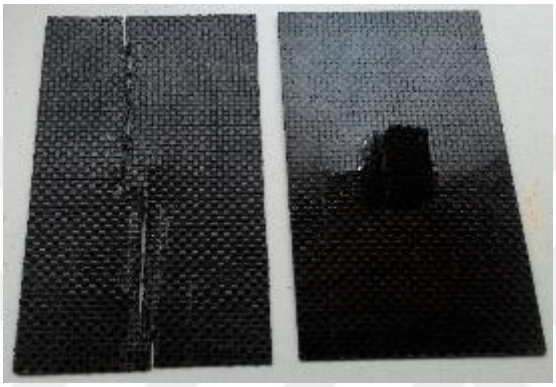

Figure 3.39: Impact response of [CCC] composites (without CNT, CNT-Epoxy dispersed).

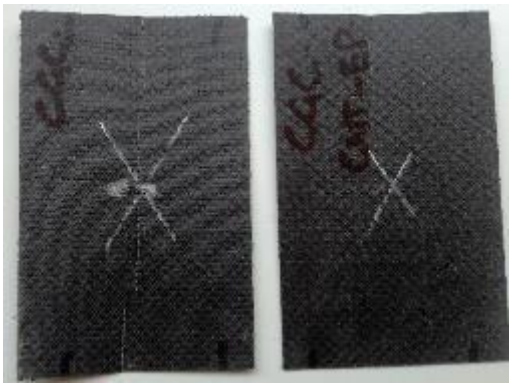

Figure 3.40: Impact response of [CGC] composites (without CNT, CNT-Epoxy dispersed).

- Maximum impact response can be seen in [GGG] composite in figure 3.42 where the impact is negligible. This is behavior was supported by the good flexural properties of the glass fiber which was enhanced through the CNTs network structure in the matrix.

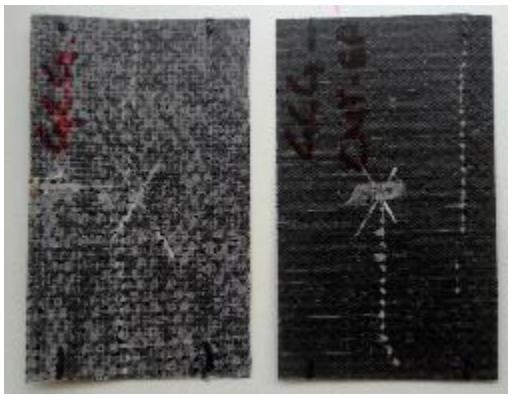

Figure 3.41: Impact response of [GCG] composites (without CNT, CNT-Epoxy dispersed).

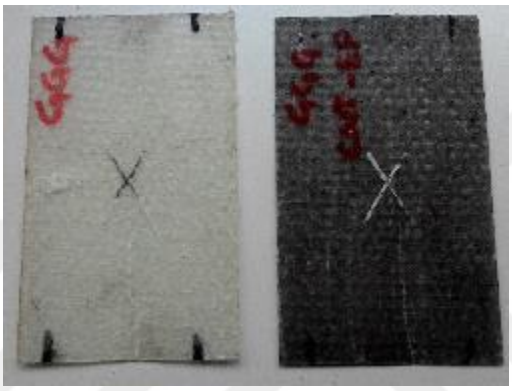

Figure 3.42: Impact response of [GGG] composites (without CNT, CNT-Epoxy dispersed).

3.3.4. Impact strength analysis for CNT-Epoxy dispersed composites

- The impact strength of the samples manufactured by CNT-Epoxy dispersion came out with significant improvement.
- [CCC] composites tend out be more stiff as compared with other samples that's why it broke out on the impact area but the damage was very limited as compared with the untreated sample.
- Introduction of glass fabric to the composites resulted in very good impact properties since it is much flexible as compared with carbon fiber. We can observe from the test specimen that the cracks appeared on the samples with glass reinforcement but it didn't break.
- The CNT-Epoxy interaction makes out a network structure which distributes the applied impact in all direction resulting in a force which avoids shearing effect to a certain extent making the composite impact absorbing ability to a certain higher limits (Tang, et al., 2013).

- The maximum improvement can be seen in the [CGC] CNT-Epoxy dispersed composites which shows close to negligible damage. The sandwiched glass reinforcement with high flexural properties and the continuous network of matrix chains with the CNTs had supported as the core impact absorber which regained the original position after the impact.

3.4. Suggestions and Practical Application of This Study

In this study, the mechanical properties of composite laminates fabricated with carbon and glass fibre grafted with MWCNTs are investigated. In view of the previous studies about the CNT reinforced composites, this experiment was carried in order to analyse the behaviour of CNT grafting on the reinforcements. The mechanical properties are determined by performing related ASTM and ISO tests.

This grafting technique can be used to impart improved tensile and flexural strength in composites where addition of weight is not required. Further to that if cost reduction is required then this technique can further be used to impart localized grafting of CNTs on the reinforcements to improve the flexural properties. Moreover the tensile properties of the carbon fibre reinforced composites can be improved with this technique which will not add weight to the final product. The functional properties of these composites are yet to be discovered which will add more interest in the applications in aeronautics and structural applications.

The enhancement of the flexural properties with negligible addition in the weight influences the importance of such materials. They can be utilized in combination with the structural materials which require an addition in the flexural strength without sacrificing the weight. The structural materials of automobiles can be incorporated with these composites in order to avoid any extra loading of metal to share high flexural strength. Safety equipment like helmets, pads, automobile bumpers are also influenced by the materials having good flexural properties which is a good area of application for such composite materials.

Since the size of the CNTs have a very important role in improving the mechanical properties of the composites, there is a certain possibility to for huge breakthrough to achieve high performance of the composites. CNTs with different sizes can be compared using different techniques to enhance the overall performance of the

composite materials. MWCNTs are highly conductive, which means that they can be used in electrical applications. The stacking behaviour of the MWCNTs would also effect the electrical properties of the material. The electrical properties of the CNTs reinforced system can influence the sensing ability of the composite material deriving various useful applications for the future demands.

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